Development of a New Electron Spin Resonance Spectroscopy

Zhe Chen

Clemson University, zhec@g.clemson.edu

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DEVELOPMENT OF A NEW ELECTRON SPIN RESONANCE SPECTROSCOPY

A Dissertation
Presented to
the Graduate School of
Clemson University

In Partial Fulfillment
of the Requirements for the Degree
Doctor of Philosophy
Electrical Engineering

by
Zhe Chen
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Accepted by:
Dr. Pingshan Wang, Committee Chair
Dr. Hai Xiao
Dr. Igor Luzinov
Dr. Eric G. Johnson
ABSTRACT

The dissertation focuses on the development of a novel electron spin resonance (ESR) spectroscopy based on an RF interferometer. The ESR spectroscopy is broadband and quantitative which is tunable over a wide frequency range, in which the RF interferometer is used to remove radio-frequency (RF) probing signals at the detector. With a planar microwave resonator (MR) the ESR spectroscopy is able to measure as low as 0.2 µg (2.8×10^{14} spins) DPPH (2,2-diphenyl-1-picrylhydrazyl) sample at a signal-to-noise ratio (SNR) of ~ 121 at room temperature. The broadband ESR operation capability is investigated with a broadband meander micro-strip line (MML) by measuring 6 µg (8.4×10^{15}) DPPH between 8 GHz and 13 GHz at a ~194 SNR with 10 kHz VNA IF. The obtained sensitivity is significantly higher than that of current broadband ESR methods. In both MR and MML cases, dispersion and absorption ESR signals at room temperature are simultaneously obtained. With MML structures, the quantification of permeability \( \mu(\omega) = \mu'(\omega) - j\mu''(\omega) \) is possible. The ESR techniques is a promising technique in the examination of magnetic particles and thin films. More work is needed to further improve ESR sensitivity and quantification accuracy.
DEDICATION

To:

My parents, Liangbing and Xihua.
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First of all, I would like to thank my advisor, Dr. Pingshan Wang, for his mentoring during my Ph.D. time in Clemson University. His advice, support and encouragement helped me overcome a lot of difficulties, which I could have never gone through without him. His dedication and enthusiasm to research have influenced me significantly.

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CHAPTER ONE
INTRODUCTION

Electron spin resonance (ESR), also called electron paramagnetic resonance (EPR) is a phenomenon in which paramagnetic materials in a magnetic field absorb microwave radiation.

ESR spectrometer is an important technique for the study of materials containing unpaired electrons in biology, chemistry and physics [1.1-1.3]. For example, it can be used to reveal the proteins' structure [1.4]. Moreover, ESR technique is an effective tool in newly developed quantum computing since it can manipulate quantum bits [1.5]. Recently, using ESR to diagnose malaria-infected human red blood cells (iRBC) is a popular topic, since an iRBC contains the malaria pigment (hemozoin), which is a byproduct of the disease formed during the intraerythrocytic growth cycle of the parasites [1.6]. Such pigment is generally a single domain crystal of magnetite (Fe₃O₄) or greige (Fe₃S₄) [1.7, 1.8] and ESR results verify the existence of Fe³⁺ [1.9].

To excite ESR, two magnetic fields are required to applied on the material under test (MUT) containing unpaired electrons [1.1]. The first is the DC magnetic field $B_0$. With the $B_0$ applied to the MUT, the unpaired electrons energy level split into two states: $\alpha$ and $\beta$, as it is shown in Figure 1, which is called Zeeman effect. The second magnetic field is the microwave magnetic field. The microwave magnetic field should be perpendicular to the DC magnetic field $B_0$. The reason for such orientation arrangement can be explained by the Bloch equations. Moreover, the electron in $\beta$ state can absorb a quantum of electromagnetic radiation energy $h\nu$ which coincides with the energy
difference between the $\alpha$ and $\beta$ states. As a result, the frequency of the microwave satisfies:

\[ h\nu = E_\alpha - E_\beta = g \mu_B |B_0| \]  

(1.1)

However, the materials we are measuring contain more than $10^{10}$ spins and if we consider the interaction between the spins with the environment, the ratio between the number of $\alpha$ spins to the $\beta$ follow Boltzmann distribution law:

\[ \frac{N_\alpha}{N_\beta} = \exp\left(-\frac{g \mu_B |B_0|}{k_B T}\right) \]  

(1.2)

where the $k_B$ is the Boltzmann constant, which is equal to $1.3806 \times 10^{-23}$ J K$^{-1}$ and $T$ is the absolute temperature [1.2].

So far, the energy level model clarifies the physical process. However, in our measurement, we are more interested in the macroscopic characteristics, which can be described by the Bloch equations [1.1].
The total magnetization is the sum of individual magnetic moment:

\[ \vec{M} = \frac{1}{V} \sum_{i}^{N} \vec{\mu}_i \]  

(1.3)

where \( V \) is the volume of the MUT. To excite the time dependent \( M \), we need the DC magnetic field \( B_0 \) and the microwave magnetic field \( B_1 \). For convenience, the \( B_0 \) is in \( z \) direction while the \( B_1 \) is in the plane of \( xy \). The equation of motion of \( M \) is:

\[ \frac{d\vec{M}}{dt} = \vec{M} \times g \mu_B \vec{B} \]  

(1.4)

By solving the differential equation, we have the Bloch equations:

\[ \frac{dM_x}{dt} = \gamma \left( \vec{M} \times \vec{B} \right)_x - \frac{M_x(t)}{T_2} \]

\[ \frac{dM_y}{dt} = \gamma \left( \vec{M} \times \vec{B} \right)_y - \frac{M_y(t)}{T_2} \]  

(1.5)

\[ \frac{dM_z}{dt} = \gamma \left( \vec{M} \times \vec{B} \right)_z - \frac{M_z(t) - M_0}{T_1} \]

where \( \gamma = g \mu_B / \hbar \) is the gyromagnetic ratio. \( T_1 \) is the spin-lattice time constant, which is related to the energy transfer to the lattice; \( T_2 \) is the spin-spin relaxation time and the energy will conserve in the spin system.

Solving the differential equations with the magnetization:

\[ M = \chi \frac{\vec{B}}{\mu_0} \]  

(1.6)

where \( \mu_m \) is the permeability of the medium, we can obtain the permeability of the material during ESR:
where $\chi^0$ is the static magnetic field magnetic susceptibility $\kappa\mu_0 g^2 \beta^2 \gamma / 4k_B T$, $\kappa=1$ for the Lorentzian lines; $\omega = g \beta B_0 \hbar$ is resonance frequency; $B_0 = 2B_1 \cos(\omega t)$ is the microwave magnetic field. $\chi'$ is the dispersive part and $\chi''$ is the absorption part [1.2]. We expect that the ESR spectrum should follow the dispersion and absorption.

Equation (1.1) suggests two ways of performing ESR experiment. One is fix the microwave frequency $\nu$ while sweeping the magnetic field $B_0$; the other is fix the magnetic field $B_0$ while sweeping the microwave frequency. In the experiments in the following chapters, we focus on the former. Nevertheless, the system we will build is versatile and it can fix the magnetic field and sweep the microwave frequency.
One of the MUT we will test is 2,2-diphenyl-1-picrylhydrazyl (DPPH), which is a standard of the position and intensity of electron paramagnetic resonance signals. If we fix the frequency at 12.3 GHz and sweep the $B_0$ we can use equation (1.6) to compute the magnetic susceptibility $\chi'$ and $\chi''$ by using $N_v \approx 2 \times 10^{27}$ spins/m$^3$ and $\tau_1=\tau_2=62$ ns [1.10], as it is shown in Figure 1.2: the $\chi'$ and $\chi''$ are so small that very sensitive detection method is needed to obtain the ESR spectrum.

To measure ESR signal with continuous wave (CW), there are mainly two major methods in current state. The first one is a field modulation technique. Aside from the DC magnetic field $B_0$, a modulation magnetic field at kHz range is applied [1.1-1.3]. As a result, the ESR signal is modulated and by a lock-in detection system, the first derivative of the absorption curve is recorded. The details of the method will be introduced in Chapter IV.

Since the ESR signal is modulated to kHz frequency range, the low frequency noise including cable and microwave connector connection, thermal effect, $1/f$ noise and others are removed. As a result, such method has a very high sensitivity. However, it also has some major drawbacks [1.11]. Since it measures the first derivative of the absorption curve, restoring the original signal requires careful calibration of the detection system. Usually, a quantitative measurement is a difficult task for the method [1.1, 1.12]. Moreover, the field modulation may distort the spectrum if the modulation field intensity is larger than the linewidth. For an unknow material, the selection of the modulation amplitude requires several try-and-errors. Also, the modulation field will generate eddy current on the resonator. Such microphonic noise becomes problematic at high-
frequencies (e.g. at terahertz) [1.13, 1.14]. Finally, the intrinsic shortcoming arises if the MUT has a broad spectrum. The sensitivity of this much will significantly decrease, because the value of the first derivative is much lower than narrow spectrum’s.

To overcome the disadvantages of field modulation technique, the direct measurement without field modulation gain its popularity these days. Typically, the $S$-parameters of the sensing device is measured by a vector network analyzer (VNA), which can reflect the absorption and dispersion of the ESR [1.15, 1.16]. This method can also use a broadband device, thus being applied to broadband and multi-frequency ESRs for many important fields of research, including the distinction between field-dependent and field-independent paramagnetic resonance processes, the study of frequency-dependent linewidth, and the differentiation between the spectra of different paramagnetic species [1.17]. These broadband structures can achieve high filling factors, but the achievable ESR sensitivity is compromised due to the absence of frequency-selectivity. For example, multiple measurements are needed for averaging in [1.14, 1.18]. And the coplanar waveguides (CPW) based ESR system worked at 1.4 K since low temperature operation significantly enhances ESR processes [1.19, 1.20].

Therefore, a non-field modulation technique which can achieve high sensitivity is strongly desired. In this work, a novel ESR spectrometer is proposed, which based on a recently proposed RF interference device that achieves high sensitivity operations while covering a very wide range of frequencies. Dielectric liquid mixtures at record low concentration levels have been successfully characterized with the technique [1.21, 1.22]
and the RF technique is further developed to address the ESR challenges mentioned above.

In Chapter II, the ESR spectrometer based on an RF interferometer is presented. Its working principles are introduced with equations and the equations are verified by the Advanced Design System (ADS) [1.23] and ANSYS Electromagnetics (HFSS) [1.24] simulations. After the simulation, the broadband ESR experiment is performed with a broadband meander micro-strip line (MML) by measuring 6 μg (8.4×10^{15}) DPPH between 8 GHz and 13 GHz at a ~194 SNR with 10 kHz VNA IF. The obtained sensitivity is significantly higher than that of current broadband ESR methods and dispersion and absorption ESR signals at room temperature are simultaneously obtained. With the MML structures, the quantification process of permeability $\mu(\omega) = \mu'(\omega) - j\mu''(\omega)$ from the measurement results is also shown.

Chapter III focuses on the sensitivity improvement for the ESR spectrometer. The idea is to substitute the transmission line with a resonant structure like a microstrip resonator (MR) or a coupling-line filter (CL). By using the resonator and filter as the sensors, the interactions between RF waves and material-under-test (MUT) can be significantly strengthened due to enhanced RF fields and longer interaction time when compared with uniform transmission lines. To simplify the process, simulations and experiments with dielectric material instead of magnetic material is used to demonstrate the idea. The results show that the MR and CL can significantly enhance RF interferometer sensitivity, e.g. by more than 5 times of frequency shift and up to 40 dB more of transmission coefficient change around 2 GHz. After proving the idea, we apply
the method to the ESR measurement. A transmission resonator working at 7.6 GHz with the interferometer can measure 0.2 μg DPPH with 0.2 μg (2.8×10^{14} spins) DPPH with a \( SNR \) of \(~121\). Moreover, a reflective resonator is also designed with the interferometer adapted accordingly to measure \( S_{11} \) instead of \( S_{21} \). The filling factor of the resonator is increased by minimizing the transmission line length on a high permittivity substrate.

To understand the contribution of the RF interferometer in the sensing process, we seek to the conventional lock-in detection method, whose sensing process is similar to the VNA but less integrated. In Chapter IV, the lock-in detection working principles are first introduced. We build the lock-in detection system working at 3 GHz. Then we incorporate the RF interferometer into the system and the ESR signal is amplified by a low noise amplifier (LNA). As a result, the signal to noise ratio is increased by 7 times due to the amplified signal against a fixed detector noise. The RF interferometer significantly increases the dynamic range and avoids non-linear effect at the mixer. With the knowledge of the lock-in detection, the working principle is extended to the VNA detection. By investigating the working principles of the VNA, it is concluded that the non-linear effect is avoided and the analog to digital conversion (ADC) precision is increased, thus benefiting the small signal detection.

Chapter V is the exploration of building high frequency sensors. Waveguides based sensors are designed, simulated and measured. Simulations and experiments prove the effectiveness of these sensors in detecting permeability and permittivity change of the MUT. However, further work is needed to understand the sensing mechanism of the waveguide sensors. Their performance and sensitivity should also be improved.
REFERENCES


CHAPTER TWO

BROADBAND ESR OPERATION

INTRODUCTION

Electron spin resonance (ESR) spectroscopy, i.e. electron paramagnetic resonance (EPR) spectroscopy, is an important technique for the study of materials in biology, chemistry and physics containing unpaired electrons [2.1]. Modulate external magnetic field ($B_{\text{ext}}$) while keeping electromagnetic radiation frequency ($f_0$) constant has been the dominant ESR instrumentation method. The approach works in synergy with high quality factor ($Q$) single-frequency reflection (1-port) resonators to achieve high ESR sensitivities [2.2].

Nevertheless, the narrow ESR operating frequency range, which is mainly limited by the high-$Q$ resonator, makes it difficult to study less conventional magnetic materials, such as those with large zero-field splitting or numerous magnetic transitions [2.3], for which measurements at a single ESR frequency might not be sufficient to elucidate the level structure [2.4, 2.5]. In order to observe field-induced spin level crossing or anticrossing, which often has unknown energy gap values, it is also desirable to have a broadband spectrometer that is tunable over a wide frequency range [2.6]. To address these problems, ESR techniques with tunable resonators [2.3, 2.6] and non-resonant devices, such as transmission lines [2.4, 2.7, 2.8], small coils [2.9], and antennas [2.10], have been reported. However, the tunable resonators are a problem when investigating processes that involve both low frequency and high $B_{\text{ext}}$ [2.4, 2.6] since large resonator sizes often limit the available $B_{\text{ext}}$; non-resonant devices usually result in compromised
ESR sensitivity [2.10] even though increasing filling factors helps make up for some of the loss [2.9]. Another challenge is quantitative ESR measurement [2.11, 2.12], which involves accurate calibration of each ESR system component and careful match consideration between standards and test samples [2.13].

Recently, we demonstrated a tunable interferometer which operates from ~ 10 MHz to ~ 40 GHz [2.14] with effective quality factors ($Q_{\text{eff}}$) up to $10^8$ [2.15]. The interferometer is effective for highly sensitive and broadband quantification of dielectric material permittivity values, i.e. $\varepsilon(\omega) = \varepsilon'(\omega) - j\varepsilon''(\omega)$, with vector network analyzer (VNA) measurements [2.16-2.18]. In this work, we show that such a tunable microwave interferometer can be modified and used with a VNA to build broadband ESR systems. High resolution tuning of the interference process enables accurate measurement of frequencies and direct measurement of ESR absorption. Thus, broadband, sensitive, and quantitative ESR measurements are obtained.

This chapter is arranged as the following. Section II describes the operation and design consideration of the broadband ESR system. Section II presents measurement results. Section IV includes discussions and conclusions.
Figure 2.1 shows a schematic of the ESR system in frequency sweeping operation. The VNA sweeps frequency for a given external magnetic field $B_{\text{ext}}$. The obtained S-parameter trace, $|S_{21}|$, will shift if a second given $B_{\text{ext}}$ induces a different MUT response. The frequency shift $\Delta f$ of $|S_{21}|_{\text{min}}$, the magnitude change of $\Delta |S_{21}|_{\text{min}}$ and $\Delta |S_{21}|_{\phi}$ are all ESR indicators. The effective quality factor of the interferometer is defined as $Q_{\text{eff}} = f_0/f_{3\text{dB}}$.

ESR SYSTEM DESIGN CONSIDERATIONS

Figure 2.1 shows a schematic of the ESR system in frequency sweeping operation.

The set-up includes an interferometer (the components connected with blue solid lines), a VNA (model R&S ZVA50 calibrated with an Agilent 85052d kit), and an electromagnet (GMW 3470) (the three components connected with green dashed lines). The interferometer consists of two broadband Wilkinson power dividers (PD), a tunable phase shifter ($\phi$) and an attenuator ($R$). Similar to the operation of the dielectric spectroscopy system [2.14], the phase shifter is used to tune the phase of RF reference wave $A_{\text{REF}} = A_1 \cos(\omega_0 t + \Phi_{\text{REF}})$, so when it is combined with the RF probing wave, $A_{\text{MUT}} = A(1+a) \cos(\omega_0 t + \phi + \Phi_{\text{MUT}})$ that is from the material-under-test (MUT) branch, the
phase difference between $A_{REF}$ and $A_{MUT}$ satisfies $\Phi_{REF} - \Phi_{MUT} = (2n-1)\pi$, $(n = 1, 2, 3...)$ at the targeted measurement frequency $f_0$. Parameter $\Phi_{MUT}$ is the phase when no ESR interaction occurs while $a$ and $\varphi$ are ESR induced magnitude and phase change, respectively. Integer $n$ is the operating harmonic number of the interferometer. At the same time, the attenuator is used to tune the amplitude of $A_{REF}$ so the two waves are well balanced to obtain very low $|S_{21}|_{min}$ values for high sensitivity ESR operations.

The interferometer arrangement in Figure 2.1 resembles that of a microwave bridge, which is widely used in ESR instrumentation for high-sensitivity measurement [2.11, 2.19]. Nevertheless, there are a few major differences, which include the use of a destructive interference process, the exploitation of high-resolution tuning (e.g. down to $\sim 10^{-4}$ dB for attenuation), and the employment of harmonic frequency operation. Specifically, the attenuator in Figure 2.1 is for sensitivity (i.e. $|S_{21}|_{min}$) tuning whereas the attenuators in a conventional bridge are used to avoid ESR saturation at the resonator and to optimize ESR signal detector performance. As a result, lower than -90 dB of $|S_{21}|_{min}$ is easily obtained for the set up in Figure 2.1 with $\sim 0$ dBm power from port 1 and at 100 Hz measurement intermediate frequency (IF). The outstanding $|S_{21}|_{min}$ enables much more sensitive measurements than the -60 dB achieved in [2.20]. Such fine tuning and direct measurement of ESR absorption have not been reported for bridge circuits [2.4, 2.21] or optical interferometer ESR detections [2.5]. Additionally, the phase shifter in Figure 2.1 is for operating frequency $f_0$ tuning whereas the phase shifter in the reference arm of a conventional ESR microwave bridge is used to ensure that the reference arm microwaves
are in phase with the ESR microwave signals when the two signals combine at the detector [2.11].

For the VNA in Figure 2.1, it functions as an ESR microwave source and signal detector, similar to the VNAs or the digital detection systems in previously reported ESR efforts [2.22-2.24]. At port 2 of Figure 2.1 (when $S_{21}$ is measured), the output of the interferometer can be approximated as

$$A_{\text{output}} = A_0 \left( a \cos \omega t + \phi \sin \omega t \right) + \Delta A_{\text{remain}} \cos(\omega t + \Delta \Phi)$$

where the first term is ESR signal $A_{ESR}$, and the second is the remaining probing wave due to non-ideal tuning. As long as $|\Delta A_{\text{remain}}|$ is much smaller than ESR signal, $|A_{\text{output}}|$ is essentially $A_{ESR}$. It is accurately and directly measured by the VNA. Since the remaining power level is $|S_{21}|_{\text{min}}$ in Figure 2.1, thus, a -90 dB value indicates that a pico-Watt ESR absorption power can be directly detected. This is equivalent to the power absorbed by ~$10^8$ spins at resonance at room temperature [2.25]. It is significantly more sensitive than the nanoscale thermocouple [2.26]. When $|\Delta A_{\text{remain}}|$ is relatively large, which is the case for broadband transmission line systems [2.4, 2.7, 2.8], the ESR signal is overwhelmed, and the VNA may suffer from nonlinearity effects due to the so-called Townes noise in submillimeter spectroscopy [2.27]. Such noise is a challenging issue for all heterodyne detectors, including the most sensitive ESR detectors [2.19]. Therefore, the new method in can help resolve ESR sensitivity, frequency coverage, and quantification problems.

The sensing device providing RF magnetic field interacting with the MUT in this experiment is a broadband microstrip meandered line (MML). The MML in coplanar waveguide (CPW) form is used in [2.21]. In the meandered part, the line width is reduced
to 100 \mu m wide and the gap between each line is 300 \mu m to reduce crosstalk [2.28]. The high impedance line behaves high inductance and produces strong magnetic field by
increasing the current density. Strong magnetic field is beneficial to the magnetic material detection. A triangle tapering connects the 50 Ω line to extend working bandwidth. The reason for the meandered arrangement is to meet the requirement of orthogonal RF magnetic fields to $B_{ext}$ for ESR interactions as it is shown in Figure 2.2 (b).

The MML is simulated in HFSS and fabricated on a 1mm thick quartz (SiO$_2$) substrate with ~ 0.35 µm gold film. The simulation and measurements results in Figure 2.2 (c) show relatively high insertion loss, which is mainly due to high MML resistance (~44 Ω measured at DC). The high DC resistance mainly comes from its long length and ununiform thin gold film. Reflections make the insertion loss even worse than the simulated results. The insertion loss will reduce $|S_{21}|$ (Figure 2.1) and corresponding ESR sensitivity since each dB of insertion loss is equivalent to 1 dB increase of device
noise figure. The RF magnetic field distribution of the MML is non-uniform between two adjacent metal lines. Thus, only a fraction of DPPH powder is involved in ESR interactions. Nevertheless, the use of MML is necessary due to the small pole areas of the electromagnet used in this work. Within 1 mm² area at the center, $B_{\text{ext}}$ variation is $\sim 0.1\%$, i.e. $\sim 3$ G for 3000 G $B_{\text{ext}}$ field. The variation further limits the amount of DPPH in effective ESR interactions.

The dielectric spectroscopy system described in [2.14] has proven that the real part $\varepsilon'$ changes the equivalent capacitance $C$ of a piece of transmission line, and it will induce $\Delta f$ in Figure 2.1. In the meantime, the imaginary part $\varepsilon''$ produces the magnitude change of the $A_{\text{MUT}}$ and $\Delta|S_{21}|_{\text{min}}$. Similarly, when ESR induces $\mu = \mu' - j\mu''$, $\mu'$ will add to the inductance $L$, while $\mu''$ contribute to the resistance $R$. So we expect $\Delta f$ follows $\chi'$, while $\Delta|S_{21}|_{\text{min}}$ follows $\chi''$.

Simulations are performed to predict the response of the interferometer in Figure 2.1. In HFSS [2.29], a 1 mm×1 mm×0.1 mm MUT slab is put at the center of the line where DPPH sample is placed, Figure 2.2 (b). To mimic ESR effects, we first tune $\mu'$ of the MUT slab from 1 to 1.008 while keeping its $\mu'' = 0$, then tune $\mu''$ from 0 to $4 \times 10^{-3}$ with $\mu' = 1$. The scattering parameters from these HFSS simulations are exported in .s2p files. A model of the interferometer in Figure 2.1 is built in Advanced Design System (ADS) [2.30] with ideal circuit elements, including power dividers, an attenuator and a phase shifter. The .s2p files are used to represent the MML when there is MUT. The arrangement of these elements is the same as that in Figure 2.1. Even if we are using the 3rd harmonic in our experiment ($n=3$ in $\Phi_{\text{REF}} - \Phi_{\text{MUT}} = (2n-1)\pi$), the phase difference of the
two branches is set to be $45\pi$ to increase the $Q_{\text{eff}}$. The parameter $|S_{21}|_{\text{min}}$ illustrated in Figure 2.1, is initially tuned to $-70\text{dB}$ with $\mu' = 1$ and $\mu'' = 0$. Conduction boundary for the gold microstrip line is used for convenience. The results are shown in Figure 2.4.

Figure 2.4 (a) and (b) indicate that $\mu'$ mainly causes $f_0$ (at $|S_{21}|_{\text{min}}$) shift and $\mu''$ produces $\Delta|S_{21}|_{\text{min}}$. However, small $\Delta|S_{21}|_{\text{min}}$ vs. $\mu'$ and $\Delta f$ vs. $\mu''$ are also observed. The
former is probably caused by $\mu'$ induced mismatches, as is observed in HFSS simulations. The latter can be attributed to propagation constant change due to $\mu''$ [2.31]. Nevertheless, the results indicate that ESR dispersion and absorption signals are obtained simultaneously, unlike in standard CW ESR and when source is locked to the cavity resonant frequency, only one of them is obtained.

**ESR EXPERIMENT**

In this experiment an iterative process is followed: First, set the range and step size of the VNA frequency sweep with a 10 kHz IF. Corresponding integration time is ~ 0.1 ms. Then, manually tune the attenuator and phase shifter in Figure 2.1 to obtain the desired initial $|S_{21}|_{\text{min}}$ at the desired initial $f_0$ while the DC magnetic field is on and at an initial value, $B_{\text{ext}}$. These parameters will be the references for the measurement. After that, the DC magnetic field will be changed to the next value automatically through a control computer, which will also record the measured $S$-parameters after each DC field change. The control computer synchronizes the frequency sweeping of the VNA and magnetic field scan. In our measurement, each sweep takes 0.054 s. Therefore, the magnetic field changes every 0.054 s and no field-modulation is used. We measure 700 data points, so it takes about 38 s to collect the whole spectrum.

A two-step broadband ESR experiment is conducted to investigate the performance of the setup in Figure 2.1 with MML. First, a frequency domain ESR is conducted. A 4.5 mg DPPH ($7.50 \times 10^{18}$ spins) is applied to the MML (Figure 2.2 (b)),
which is directly connected to the VNA without the interferometer arrangement. Measurements are conducted with $B_{\text{ext}}$ fixed while the VNA frequency swept. Figure 2.5 (a) and (b) show some typical results. Clear ESR signals are obtained, with symmetric $\Delta |S_{21}|$ and $\Delta \angle S_{21}$, as expected. Secondly, the MML is incorporated into the interferometer in Figure 2.1, with the same VNA setup but 6 µg DPPH (8.4×10$^{15}$ spins).

Measurements at 8.5 GHz, 9.5 GHz, 10.3 GHz, 11.3 GHz and 12.3 GHz are performed by tuning the phase shifter in the reference branch, as shown in Figure 2.6 (a). Figure 2.6 (b) shows typical $|S_{21}|$ at 12.3 GHz and different $B_{\text{ext}}$. Figure 2.6 (c) and (d) show the $\Delta |S_{21}|_{\text{min}}$ and $\Delta f$ vs. $B_{\text{ext}}$. Table 2.1 summarizes the measurement results at these five frequency points, which are arbitrarily selected to illustrate the broadband operation capability of the system. Any other frequency point between 8.0 GHz and 13 GHz can be measured by tuning the phase shifter.
Figure 2.6 (a) Tuning system operating frequency with the phase shifter in Figure to 8.5 GHz, 9.5 GHz, 10.3 GHz, 11.3 GHz and 12.3 GHz (marked with circles). The unmarked minima on the curves indicate different interferometer harmonic frequencies, which can also be used for ESR measurements. (b) Measured $|S_{21}|$ under different $B_{\text{ext}}$ with MML and DPPH. (c) Measured $\Delta|S_{21}|_{\text{min}}$ and (d) $\Delta f$ under different $B_{\text{ext}}$ with MML.

Table 2.1 shows that the maximum signal magnitude $\Delta|S_{21}|_{\text{min,0}}$ and frequency shift
Δf₀ increase with frequency. However, the line width ΔB is not obviously frequency-dependent. Nevertheless, the observed ΔB is larger than the reported 2 G [2.32], probably due to B_{ext} non-uniformity.

From Table 2.1, the g-factor of DPPH can be obtained via \( h f_0 = g \mu B_{ext,0} \). The average value over the five frequency points is \( g_{MM} = 2.0225 \). The single frequency resonator measurement at 7.6 GHz gives \( g_{MR} = 2.0209 \). The values deviate from the reported \( g_{DPPH} = 2.0036 \) by 0.94% and 0.86%, respectively. The main reason for the deviation is likely from the magnetic field calibration error. A Gaussmeter is used for the calibration, but its exact location has uncertainties.

**SIGNAL TO NOISE RATIO AND MINIMUM NUMBER OF DETECTABLE SPINS**

Signal to noise ratio (SNR) is an important parameter characterizing the quality of an ESR spectroscopy. The signal is the measured ESR signal while the noise sources are more complicated. Usually, thermal noise from the resistance of the sensing devices is

| \( f_0 \) (GHz) | \( B_{ext,0} \) (mT) | \( \Delta B \) (G)\(^a\) | \( \Delta |S_{21}|_{min,0} \) | \( \Delta f_0 \)\(^b\) |
|----------------|-------------------|-----------------|-----------------|-------------|
| 8.5            | 299.612           | 4.49            | 1.22×10⁻⁴       | 0.42 MHz    |
| 9.5            | 318.218           | 4.13            | 1.40×10⁻⁴       | 0.64 MHz    |
| 10.3           | 365.576           | 4.63            | 1.73×10⁻⁴       | 0.76 MHz    |
| 11.3           | 399.098           | 4.09            | 2.02×10⁻⁴       | 0.85 MHz    |
| 12.3           | 437.457           | 4.37            | 2.40×10⁻⁴       | 0.93 MHz    |

\(^a\) It is the full width at half of \( \Delta |S_{21}|_{min,0} \) as indicated in Figure 2. (c). \(^b\) \( \Delta f_0 = \Delta f_{max} - \Delta f_{min} \) which is indicated in Figure 2. (d).
considered as the limiting noise [2.12, 2.32]. However, in practice, the noise from the detector is usually much higher than the thermal noise, thus making the signal to noise ratio smaller than the ideal case [2.32]. In our measurement, aside from the thermal noise and the detector’s noise, mechanical vibrations of the attenuator, phase shifter and cables will result in higher noise. As a result, it makes more sense to directly read the SNR from the measurement results.

The specific expression of the noise is, 

\[
N_s = \frac{1}{\sqrt{2}} \frac{\sum_{n=1}^{N} (\Delta |S_{21}|_{\text{min},n} - \Delta |S_{21}|_{\text{min,avg}})^2}{\sqrt{\sum}}
\]  

(2.2)

where

\[
\Delta |S_{21}|_{\text{min,avg}} = \frac{1}{N} \sum_{n=1}^{N} \Delta |S_{21}|_{\text{min},n}
\]  

(2.3)

with \( N = 51 \). Then, the \( \text{SNR} = \Delta |S_{21}|_{\text{min,0}}/N_s \). The measurement in Figure 2.5 gives for \( N_s = 0.79 \times 10^{-4} \) with \( \text{SNR} = 234 \), and in Figure 2.6 (c) for \( N_s = 1.22 \times 10^{-6} \), \( \text{SNR} = 194 \). These values are favorably compared against the 1.92 in [2.12], where the used materials have \( 1.65 \times 10^{17} \) spins with \( \chi'' = 4.08 \times 10^{-7} \) and a 600 kHz detection bandwidth was used. The two MML ESR measurements achieve similar SNR despite 750 times more DPPH materials used for the conventional VNA-based measurements. The difference illustrates the performance enhancement of the interferometer in Figure 2.1.

Like the conventional ESR, in our measurement, the VNA noise floor (NL) is the detector noise in amplitude. The used VNA is a Rohde and Swartz (R&S) ZVA 50 which operates from 10 MHz to 50 GHz. When IF = 10 Hz, the noise floor (amplitude noise) at our measurement frequencies is -115 dBm [2.33]. If we set the IF to be 10 kHz, the noise
The signal power we use is 6 dBm (for resonator experiment) and the obtained -91 dB $S_{21}$ magnitude should be the noise signal level. Our maximum transmission signal $|S_{21}|$ is at -85 dBm (A factor-of-10 decrease in IF BW will reduce the noise floor by 10 dB).

Figure 2.7 (a) Measured $|S_{21}|$ under different $B_{\text{ext}}$ with MML and DPPH with IF = 1 kHz. (c) Measured $\Delta|S_{21}|_{\text{min}}$ and (d) $\Delta f$ under different $B_{\text{ext}}$ with MML with IF = 1 kHz.
70 dB. Therefore, it agrees with our SNR of ~100. By reducing the IF bandwidth to 1 kHz, less noise is observed in Figure 2.7 and the SNR is increased by ~2.8 times than that with IF = 10 kHz.

SNR is an indicator of the measurement quality. However, to quantify the sensitivity, the parameter called minimum number of detectable spins is used, which can be expressed as,

\[ N_{\min} = \frac{N_{\text{spin}}}{\text{SNR} \, \Delta H_{pp} \sqrt{\text{ENBW}}} \]

where \( N_{\text{spin}} \) is the number of spins in the MUT, \( \Delta H_{pp} \) the linewidth of the ESR spectrum and \( \text{ENBW} \) referring equivalent noise band width of the detector. Here we use the value of the IF bandwidth for this value. So in this broadband measurement, the achieved \( N_{\min} \) = 3.4×10^{12} (\( Q=1 \)) which is less sensitive than that of 5×10^{10} in [2.34]. The reason for the higher sensitivity is that a resonator with \( Q = 60 \) is used.

MULTIPLE-SCAN ESR

In [2.7, 2.12] for the direct measurement without a microwave bridge, the ESR measurement is repeated by thousands of times and the random noises are filtered through summation. As we have proved, the sensitivity of the ESR measurement is increased by 750 times compared with the direct measurement. Therefore, to achieve the same level of sensitivity, significantly less measurement times are required. This section is going to demonstrate the idea of multi-scan ESR with the RF interferometer.
Moreover, even if decreasing the IF can increase the SNR, each measurement

Figure 2.8 (a) Measured $\Delta|S_{21}|_{\text{min}}$ and $\Delta f$ under different $B_{\text{ext}}$ with MML with measurement times $N = 1$ at 10 GHz with $\sim 1 \mu g$ DPPH. (b) Measured $\Delta|S_{21}|_{\text{min}}$ and $\Delta f$ under different $B_{\text{ext}}$ with MML with measurement times $N = 200$ with $\sim 1 \mu g$ DPPH.

Moreover, even if decreasing the IF can increase the SNR, each measurement
takes longer time. Consequently, the RF interferometer may suffer from drifts due to the cable connections and mechanical vibrations of the phase shifter and attenuator. The multi-scan ESR can solve the problem if each measurement is conducted with a higher IF but a shorter time. After each measurement the system is re-tuned. To realize this frequent tuning, a programmable attenuator (TELEMAKUS TEA13000) should be used to save

Figure 2.9 (a) SNR vs. measurement times of the magnitude $\Delta|S_{21}|_{\text{min}}$ at 10 GHz. (b) SNR vs. measurement times of the frequency shift $\Delta f$ at 10 GHz.
tedious labor work. The measurement steps are the same to the previous ones, except the automated tuning after each measurement and the summation of all of the measurement.

Figure 2.8 shows the measurement results. (a) is the one-time measurement and (b) is the summation of 200 measurements. The SNR vs. the measurement times are illustrated in Figure 2.9 and the SNR increases with the measurement time in both magnitude and frequency shift.

The summation of several measurements is equivalent to decrease the IF bandwidth, but it is more practical because it overcomes the potential drifts during the long measurement time. The current issue with the system is the extra noise from the programmable attenuator, which is larger than that of the mechanical attenuator from the measurement results.

QUANTITATIVE PERMEABILITY CALCULATION

The measurement of the spectroscopy is quantitative, so it is possible to abstract the magnetic permeability from the measurement results. To obtain the $\Delta|S_{21}|$ and $\Delta\angle S_{21}$ of the MML from the measurement results, we need to start from the basic working principles of the RF interferometer to build the relationship between $\Delta|S_{21}|_{\text{min}}$ and $\Delta|S_{21}|$ and $\Delta f$ and $\Delta\angle S_{21}$ by a node analysis in Figure 2.10.

The measured $S_{21}$ can be expressed by its definition as [2.31],

$$S_{21} = \frac{V_1 + V_2}{V_i}$$

$$= \frac{\sqrt{2}}{2} A_1 e^{j\phi_{\text{REF}}} + \frac{\sqrt{2}}{2} A_2 |1 + \delta| e^{j(\phi_{\text{REF}} + \angle \delta)}$$

(2.5)
The operation point of the RF interferometer satisfies

\[ \phi_{\text{REF}} - \phi_{\text{MUT}} = (2n + 1)\pi, n = 0, 1, 2, \ldots \]
\[ A_1 \approx A_2 \]  

(2.6)

At the initial state by tuning the attenuator and phase shifter \[ |S_{21}|_{\text{min,ini}} = \sqrt{2}/2 |A_1 - A_2| \] and with the ESR signal \[ |S_{21}|_{\text{min,MUT}} = \sqrt{2}/2 |A_1 - (A_2 + \delta)| \]. Therefore, we have

\[ \Delta|S_{21}|_{\text{min}} = 20 \log \left( \frac{|A_1 - (A_2 + \delta)|}{|A_1 - A_2|} \right) \]

(2.7)

TEM mode propagates in the RF system and the phase follows a linear relationship. According to the definition of the group delay \[ T = -d\phi/d\omega \], with the linear phase assumption, the phase of each branch can be expressed as,

\[ \phi = -T\omega + b \]

(2.8)

and the phase difference between the two branches of the RF interferometer is,
If an extra phase $\angle \delta$ is introduced by ESR, the frequency shift is

$$\Delta f = f_0 - f'_0 = \angle \delta / (T_{REF} - T_{MUT})$$  \hspace{1cm} (2.10)$$

The equations describing the operation principles of the RF interferometer can be verified by the ADS simulations. The schematics in the ADS environment are shown in Figure 2.11. Figure 2.11 (a) simulates the outputs caused by the $\Delta |S_{21}|$ of the sensing device. The attenuator (annotated) mimics such changes and the value of such attenuation is compared with the output $\Delta |S_{21}|_{\text{min}}$ quantitatively to verify the equations. Similarly,
considering the $\Delta S_{21}$-$\Delta f$ relation, the phase is set by the annotated phase shifter. Also, since the group delay is included in the equations, a piece of delay line with known group delay substitutes the 180° transmission line in Figure 2.11 (a).

The results are shown in Figure 2.12 and the values of $\Delta|S_{21}|$ and $\Delta|S_{21}|_{min}$ and $\Delta S_{21}$ and $\Delta f$ are recorded in Table 2.2, by which equation (2.7) and (2.10) are verified.

When probing waves propagate along the reference line and MML line in Figure 2.1, the measured signal can be described as

$$S_{21} = \Delta A_{\text{rem}} e^{-j\phi} + \frac{A}{\sqrt{2}} e^{-j(\delta \alpha \tau + j \delta \beta) L}$$  \hspace{1cm} (2.11)

where the first term on the right is the phaser form of the remaining component in (1), $L$ is the line length on which MUT is uniformly distributed, $\delta \alpha$ and $\delta \beta$ are MUT induced small attenuation and phase propagation constants, respectively. When a resonator is used, strong frequency dependent signal transmission is induced by the resonator itself. Thus, differentiate MUT effects from resonator induced changes may be very involved. Therefore, we will limit our following discussions to transmission line sensing structures.

The DPPH drop on the MML’s sensing zone in Figure 2.2 (b) can be approximated as a 1.6 mm×2 mm rectangle. It covers six microstrip lines and the total length is $L = 1.6 \text{ mm} \times 6 = 9.6 \text{ mm}$. The height of the DPPH drop is estimated to be $h = m_{\text{DPPH}}/\rho S = 1.3 \mu m$, with $\rho$ the density of DPPH 1.4 g/cm³.
Since the DPPH only occupies the surface of the microstrip line and most of the

Figure 2.12 (a) Simulation of $\Delta |S_{21}|_{\min}$ vs. $\Delta |S_{21}|$. (b) Simulation of $f_0$ vs. $\angle \Phi$.

Table 2.2 Summarized simulation results in Figure 2.12

| $\Delta |S_{21}|$ (dB) | $\Delta |S_{21}|$ | $\Delta |S_{21}|_{\min}$ (dB) | $\Delta \angle S_{21}$ (degree) | $\Delta f$ (MHz) |
|-----------------|-----------------|--------------------------|-----------------|----------------|
| 0.01            | 8.14x10^{-4}    | 4.07x10^{-4}             | 1°              | 11             |
| 0.02            | 16.26x10^{-4}   | 8.13x10^{-4}             | 2°              | 22             |
| 0.03            | 24.38x10^{-4}   | 12.19x10^{-4}            | 3°              | 33             |

Since the DPPH only occupies the surface of the microstrip line and most of the
RF fields are free from it, shown in Figure 2.13, a filling factor $q_{m,l}=M_1/M_2$ [2.35] should be considered, where $M_1$ is the field flux that passes through DPPH, and $M_2$ the total field flux.

$$M_{1,2} = \iint_{S_{1,2}} H(y, z) \, dy \, dz$$

(2.12)

Using numerical integration with fields from HFSS [2.29] simulation, the integration yields $q_{m,l} = M_1/M_2 = 0.0065$. The complex propagation constant of a microstrip line is

$$\gamma = \omega \sqrt{\varepsilon_r \varepsilon_0 \mu_0 \left( \mu_{\text{eff}} - j \mu_{\text{eff}}' \right)}$$

$$= \omega \sqrt{\varepsilon_r \varepsilon_0 \mu_0 \left( 1 + \chi_{\text{eff}} - j \chi_{\text{eff}}' \right)}$$

(2.13)

With MUT induced additional attenuation constant as [2.31]

$$\delta \alpha = \frac{1}{2} \omega \sqrt{\varepsilon_r \varepsilon_0 \mu_0 q_{m,l} \chi''}$$

(2.14)

For $\chi'$, it induces propagation constant change

$$\delta \beta = \frac{1}{2} \omega \sqrt{\varepsilon_r \varepsilon_0 \mu_0 q_{m,l} \chi'}$$

(2.15)

Corresponding extra phase is $\Delta \phi = \Delta \beta L$, which can be described as [2.36]

$$\Delta \phi = (T_{\text{MUT}} - T_{\text{REF}}) \Delta f$$

(2.16)

where $T_{\text{MUT}}$ and $T_{\text{REF}}$ are the slopes of $\angle A_{\text{MUT}}$ and $\angle A_{\text{REF}}$ vs. $f$, respectively. The slopes can be obtained by measuring the MUT and REF branch. In our experiment, $T_{\text{MUT}} - T_{\text{REF}} = 1.103$ rad/GHz at 12.3GHz.
Figure 2.14 shows the calculated DPPH $\mu'$ and $\mu''$ with the data from Table 2.1. The results agree with theoretical values reasonably well. The theoretically values are obtained from (2b) and (2c) with $\tau_2 = 2/\omega_2$ and $\omega_2 = \Delta B g \beta / \hbar$. The discrepancies are likely caused by (i) filling factor $q_{m,l}$ errors due to DPPH thickness and uniformity disparities,
and (ii) cross effects of $\chi'$ and $\chi''$ on $\Delta f$ and $\Delta |S_{21}|_{\text{min}}$ as shown in simulations and measurements results.

The obtained $\chi''$ can be used to estimate ESR power. For a $\eta = 0.0065$ filling factor, a $Q_L = 1$ and $Z_0 = 50$ $\Omega$ MML, and a $P = 0.83$ mW probing power, the ESR voltage,

$$V_x = (1/2) \chi'' \eta Q_L \sqrt{Z_0 P}$$

(2.17)

is $\sim 3.78 \times 10^{-5}$ V [2.11, 2.12], which corresponds to a power of $2.85 \times 10^{-11}$ W. The power agrees with direct VNA measurement, $7.41 \times 10^{-11}$ W (-72.9 dBm), reasonably well. The calculated voltage is $\sim 100$ times higher than the voltage in equation G.12 in [2.11], mainly due to smaller $\chi''$ therein.

**DISCUSSIONS AND CONCLUSIONS**

Compared with other broadband methods, the approach in Figure 2.1 achieved much higher sensitivity. For instance, 3 mg DPPH was used in [2.10] to achieve a similar SNR at low temperature, while Figs. 5 and 7 used 0.2 and 6 $\mu$g DPPH, respectively, at room temperature. Nevertheless, compared with field-modulation method, the microresonator based ESR used 0.5 $\mu$g DPPH and obtained a 30000 $\text{SNR}$ (single frequency, room temperature) [2.32], which is $\sim 100$ times better than our results. Their reported sensitivity is also comparable to that of a commercial system (operates at low temperature).

However, the method in Figure 2.1 has the potential to achieve a sensitivity comparable to or better than that in [2.32]. Higher frequency [2.37] and higher $Q$ [2.34] will result in higher sensitivities proportionally. $\sim 4$ G linewidth is observed in Figure 2.6.
(b) while a 2 G linewidth is reported in [2.32]. Additionally, operate the interferometer at fundamental frequency rather than 3\textsuperscript{rd} harmonic could further improve sensitivity by a factor of \textasciitilde 5 [2.36]. Moreover, a 10 kHz VNA IF is used to obtain Figure 2.6 (a) and (c) while a locking time constant of 300 ms is used in [2.32]. With a smaller IF, our SNR can be further improved. These issues will be addressed in future design for better ESR sensitivity operation. Furthermore, the used VNA in our experiment is not specifically designed for ESR measurement. Its noise performance is likely inferior to the electronic systems of an ESR instrument. For instance, our used R&S ZVA50 has an amplitude noise floor of -115 dBm when IF=10 Hz [2.33] and a phase noise of \textasciitilde -100 dBc/Hz at 100 kHz offset [2.38]. Nevertheless, much better VNA noise performance is achievable with better signal sources and receivers. As a result, the sensitivity of the ESR method in Figure 2.1 can be further improved.

Compared with conventional ESR methods [2.11], the effects of MUT dielectric properties, including dielectric losses, can be tuned out with the phase shifter and attenuator in Figure 2.1. So they do not interfere with sensitive and accurate ESR measurements. Additionally, the method in Figure 2.1 can be further developed for frequency-domain ESR operations and helps address some of the technology issues in current frequency domain efforts, including the violation of “transfer of modulation” [2.10, 2.39-2.41]. Thus, it enables the studies of materials which have $B_{\text{ext}}$-dependent properties. Besides, the quantitative ESR is also promising to be a highly sensitive ferromagnetic resonance (FMR) method to examine the dynamic properties of magnetic particles and thin films, among others [2.42-2.44].
The ESR signals and sensitivity discussed above involve solid DPPH sample and room temperature operation. For practical ESR applications, however, concentration sensitivity of liquid samples and low temperature operation are also important considerations. Linewidth broadening of liquid solutions often require more materials for detection. Limited analyte solubility means a large volume sample. However, the improved absolute ESR sensitivity will benefit the concentration sensitivity proportionally. At the same time, the structures in Figure 2.2 (a) are low temperature compatible with potentially better performance due to lower loss.

In summary, the proposed broadband ESR technique utilizes a destructive interference process to remove strong RF probing signals at the output port, eliminates potential nonlinearity effects, and achieves high sensitivity operations. Tunable phase shifters and attenuators are incorporated for convenient sensitivity and operating frequency tuning. A meandered microstrip line are designed and built to demonstrate high-sensitivity and broadband ESR operations with DPPH powder samples at room temperature. Both absorption and dispersion ESR signals as well as frequency dependent permeability values are obtained. The results show that the new ESR method is promising to help address the sensitivity, frequency coverage and quantification difficulties of current non-conventional ESR techniques.

REFERENCES


CHAPTER THREE

ESR SENSITIVITY IMPROVEMENT

INTRODUCTION

Radio frequency (RF) sensors are actively investigated for various scientific studies and practical applications, such as characterizing aqueous solutions [3.1] and organic liquids [3.2], detecting and identifying particles and cells [3.3], analyzing chemical/biological materials [3.4, 5], including protein [3.6] and DNA [3.7] molecules, sensing displacements [3.8], monitoring the humidity of agricultural products [3.9], and civil engineering materials [3.10]. To achieve high sensitivity and obtain rich information about material-under-test (MUT) have been the main challenges in these efforts, which often depend on measuring MUT dielectric properties. The newly developed tunable interferometers [3.11, 3.12] aimed at solving these problems. An effective quality factor of $3.8 \times 10^6$ and a frequency tuning range from ~ 20 MHz to 38 GHz have been reported [3.11]. Approximately 50 DNA molecules in ~ 1 nL water have been measured with such interferometers. DNA molecules and their damaged counterparts can be differentiated [3.13]. The performance of these interferometers is superior to other types of microwave and RF sensors, including those based on whispering gallery mode resonators [3.14], as discussed in [3.15].

Nevertheless, the high sensitivity comes with a price, i.e. the signal detection instrument must have a large dynamic range (DR). For instance, the network analyzer in [3.11] needs to operate at a DR better than 120 dB. As a result, environmental interference, such as mechanical vibrations and scattered RF radiations, can destabilize
interferometer operations. Thus, it is of great interest to explore new techniques to relax the large dynamic range requirement of the interferometers and enhance system robustness.

Recently, we proposed to use low-pass and band-pass filters to boost interferometer sensitivity by achieving stronger interactions between RF probing fields and MUT [3.16]. For a given RF probing power and MUT volume, the interactions are determined by the interaction time ($\tau_i$) and the local RF field intensity, i.e. the magnitude of electric field $E_{rf}$ and/or magnetic field $B_{rf}$. The work in [3.16], probably also in [3.3], has shown that filters are effective in reducing RF wave velocities and enhancing local RF field intensities through manipulating the spectrum and dispersion relationships of electromagnetic structures. Additionally, it is shown that the RF interferometers are very flexible in terms of their sensing structures [3.16]. Therefore, filters are promising to help further relax the need for large DR operations of interferometers. Nevertheless, the filter group delay effects are not analyzed and interferometer sensitivity tuning effects are not studied in [3.16]. In this work, we will address these issues with a simple and new coupled-line (CL) filter.

Furthermore, resonators are well-known examples for concentrating and separating $E_{rf}$ and $B_{rf}$ fields [3.17, 3.18]. Longer $\tau_i$ is also used to explain the high-sensitivity detection of single nanoparticles with high-$Q$ micro optical resonators [3.19]. In this work, we will show that a micro-strip line resonator (MR) with longer propagation delay $T_i$ significantly improves interferometer sensitivity. Different from the interferometer in [3.20] where a transmission line resonator is used, our interferometer
performs summation operations with a passive device at the output port instead of multiplication operations. Thus, our interferometer detects both magnitude and phase changes caused by MUT. Additionally, both sensitivity and operating frequencies can be conveniently tuned for our interferometer.

Since the slow wave effect induced by a resonant structure can improve the detection sensitivity, it is natural for us to apply this technique to the ESR measurement. Therefore, the resonator used in the dielectric measurement is modified to work at higher frequency and the semi-ring is used as the sensing zone where the high current density generates strong magnetic field. Aside from the sensitivity improvement, such resonant structure can also separate the electric fields and the magnetic fields, and the dielectric loss will not affect the performance of the resonator. Unlike conventional cavity resonator operating in transverse electric mode (TE), both of whose longitude and transverse dimensions determine the working frequency, the effective dielectric constant and the longitude dimension of such planar resonator working in transverse electromagnetic mode (TEM) determine the working frequency. The dimension of the resonator can be significantly reduced if a high dielectric constant substrate is used.

The rest of the chapter is arranged as follows: section II describes the design considerations and RF properties of a coplanar resonator and a coupled line filter; section III presents the measured RF interferometer sensitivity and illustrates the efficacy of the proposed approach; section IV shows the application of the resonator to the ESR measurement. The resonator can be transmission or reflective form, so can the interferometer. Section V concludes the chapter.
SENSING SYSTEM DESIGN

Figure 3.1 (a) illustrates a schematic of the RF interferometer. Figure 3.1 (b) is a photo of the interferometer under test, where the VNA, phase shifters, attenuators and the devices are marked. The working principle of the interferometer has been introduced in [3.11]. Coplanar waveguides (CPW), which have higher sensitivity and broader bandwidth than micro-strip lines [3.21], have often been used as the sensing structures to generate RF fields and interact with MUTs. The quasi-TEM mode RF fields are non-dispersive with a field distribution and intensity well defined by CPW geometries. Thus, CPW properties, including group delay, field intensity and the corresponding interferometer sensitivity, will be used as references for the MR and CL discussion below. The geometrical lengths of the MR, CL and CPW as well as the gap spacing are the same for reasonable comparison.

The arrangement in Figure 3.1 indicates that the interferometer will operate at a frequency \( f_0 \), at which the phase difference between reference (REF) branch and MUT branch satisfies \(|\Delta \Phi|=(2n-1)\pi, n=1, 2, 3, \ldots\) For \( n=1 \) and \( m \), the interferometer is said to operate at fundamental and \( m^{th} \) harmonic frequency, respectively. The phase shifts of the resonators and filters need to be included in \(|\Delta \Phi|\). At the same time, lower insertion loss of the MR and CL obviously helps achieve higher sensitivities.

A planar split ring resonator, originally devised in [3.22], is chosen in this work. Edge coupling, instead of end coupling, as shown in Figure 3.2 (a), is exploited to achieve lower insertion loss at our targeted frequency (~ 2 GHz). The design in [3.22] is to generate strong RF magnetic fields in the ring area for paramagnetic material
Figure 3.1 (a) A schematic of the RF interferometer. Attenuator $R$ is mainly used for sensitivity control while phase shifter $\Phi$ for frequency tuning. Other power splitting devices, such as quadrature hybrids [3.12], can be used to replace the power dividers. A polydimethylsiloxane (PDMS) slab is used as MUT for MR and CL evaluation. (b) A photo of the interferometer with a CPW connected to the MUT branch.

characterizations. Here, we modify the design for local RF electric field enhancement
and group delay manipulation to boost the interactions between RF-electric fields and dielectric materials. PDMS, a material commonly used for biomedical applications with estimated permittivity ($\varepsilon' = 2.33$ and $\tan\delta = 0.0015$) and easily controlled dimensions, is used as a sample MUT to test and evaluate MR and CL effects on sensitivity improvements. Reasonable sensor outputs, from both CPW based comparison setup and filter/resonator-based setups, can be easily obtained for fair comparison.

The arrangement in Figure 3.1 indicates that the interferometer will operate at a frequency $f_0$, at which the phase difference between reference (REF) branch and MUT branch satisfies $|\Delta \Phi| = (2n-1)\pi$, $n=1, 2, 3, \ldots$ For $n=1$ and $m$, the interferometer is said to

![Diagram](image1)

Figure 3.2 (a) A layout of the MR, where $L=55.3$ mm, $L_1=23$ mm, $L_2=22$ mm, $L_3=16.9$ mm, $W=2.3$ mm, $G=0.2$ mm, $R_1=1.8$ mm, $R_2=1.0$ mm (b) The cross section view of a piece of coupled lines. (c) An equivalent circuit of the CL per unit length. (d) A layout of the CL where $L=26.5$ mm, $W=0.4$ mm, $G=0.2$ mm. The overall dimension of MR/CL/CPW in Figure 3.1(b), $L'=55.3$ mm, $W'=35$ mm.
operate at fundamental and $m^{\text{th}}$ harmonic frequency, respectively. The phase shifts of the resonators and filters need to be included in $|\Delta \Phi|$. At the same time, lower insertion loss of the MR and CL obviously helps achieve higher sensitivities.

Table 3.1 Broadband measurements of $S_{21}$ changes (magnitude and phase) induced by PDMS slabs at the targeted frequencies.

| $f$ [GHz] | Tested Device | $\Delta |S_{21}|$ [dB] | $\Delta \angle S_{21}$ [degree] | PDMS Size |
|-----------|---------------|------------------|-------------------------------|-----------|
| 1.925     | MR            | 0.28             | -1.8                          | 1         |
|           | CPW           | <0.01*           | -0.2                          |           |
|           | MR            | 0.83             | -5.0                          | 2         |
|           | CPW           | 0.01             | -0.5                          |           |
|           | MR            | 1.74             | -11.3                         | 3         |
|           | CPW           | 0.02             | -1.0                          |           |
| 1.9       | CL            | 0.01             | -1.5                          | 1         |
|           | CPW           | <0.01*           | <0.1*                         |           |
|           | CL            | 0.01             | -3.1                          | 2         |
|           | CPW           | <0.01           | -0.1                          |           |
|           | CL            | 0.02             | -6.5                          | 3         |
|           | CPW           | <0.01*           | -1.1                          |           |
| 2.2       | CL            | -0.05            | -1.4                          | 1         |
|           | CPW           | <0.01*           | -0.2                          |           |
|           | CL            | 0.05             | -2.8                          | 2         |
|           | CPW           | <0.01*           | -0.6                          |           |
|           | CL            | 0.07             | -6.3                          | 3         |
|           | CPW           | 0.01             | -1.1                          |           |
| 2.5       | CL            | 0.02             | -1.2                          | 1         |
|           | CPW           | <0.01*           | -0.2                          |           |
|           | CL            | 0.07             | -2.8                          | 2         |
|           | CPW           | <0.01*           | -0.7                          |           |
|           | CL            | 0.17             | -6.6                          | 3         |
|           | CPW           | <0.01*           | -1.2                          |           |

* Results are not repeatable
Figure 3.3 (a) Simulated (solid line) and measured $|S_{21}|$ of three devices, MR (Blue), CL (Red) and CPW (Green), without (dash line) and with (dot line) a PDMS slab (4.1×7.0×1.7 mm). (b) Simulated and measured $\beta-f$ diagram of MR, CL and CPW with/without PDMS slab. (c) Measured group delay $\tau_i$ of each device. The results agree with simulated group delay $\tau_i$ (not shown). The ripples on MR curves are probably due to reflections.
A planar split ring resonator, originally devised in [3.22], is chosen in this work. Edge coupling, instead of end coupling, as shown in Figure 3.2 (a), is exploited to achieve lower insertion loss at our targeted frequency (~ 2 GHz). The design in [3.22] is to generate strong RF magnetic fields in the ring area for paramagnetic material characterizations. Here, we modify the design for local RF electric field enhancement and group delay manipulation to boost the interactions between RF-electric fields and dielectric materials. PDMS, a material commonly used for biomedical applications with estimated permittivity ($\varepsilon' = 2.33$ and $\tan\delta = 0.0015$) and easily controlled dimensions, is used as a sample MUT to test and evaluate MR and CL effects on sensitivity improvements. Reasonable sensor outputs, from both CPW based comparison setup and filter/resonator-based setups, can be easily obtained for fair comparison.

Figure 3.2 (b) [3.23] shows the cross-section view of the coupled lines in (a). Figure 3.2 (c) is its equivalent circuit. Strong electric field coupling between the two edges of the coupling lines, described by capacitor $C_{12}$ [3.23] in Figure 3.2 (c), indicates strong RF-MUT interaction potentials. Thus, we expect the PDMS slab that covers the coupling edge shown in Figure 3.2 (a) will induce large change of the effective permittivity $\varepsilon_{\text{eff}}$. Hence, the interferometer sensitivity will be significantly boosted.

A simple one-stage coupled line filter, shown in Figure 3.2 (d), is also designed to obtain slow waves over a broader frequency range. The length of the coupled lines is $\lambda/4$ at 2 GHz. Narrow lines and narrow gaps are employed to achieve better coupling intensity and to improve interferometer sensitivities.

Figure 3.3 (a) shows the simulated and measured $|S_{21}|$ of the designed MR and
CL, which are fabricated with RT/Duroid 5870 laminates. The results of a comparison CPW, 1.6 mm wide signal line and 0.2 mm gap between signal line and ground, are also shown. The simulations are conducted with high frequency structural simulator (HFSS) [3.24]. It shows that (i) simulation results agree with measurement results reasonably well in all cases, (ii) for the MR, its quality factor is ~20, calculated from $f_c/\Delta f_{3\text{ dB}}$. Larger insertion loss, ~-2 dB at $f_0=2$ GHz, exists. The loss mainly comes from weak coupling, (iii) for the CL, its $|S_{21}|$ indicates a wider pass-band than that of the MR, (iv) the addition of a PDMS slab only affect $|S_{21}|$ slightly, as summarized in Table 3.1.

Figure 3.3 (b) shows the propagation constants, $\beta$, obtained from HFSS simulations and measurements. They agree with each other well. The introduction of PDMS slab into the structures only affects $\beta$ slightly, as indicated by the phase changes at the targeted frequency points in Table 3.1. Figure 3.3 (c) shows the measured group delays, which is proportional to the slope of the measured $\beta$ in Figure 3.3 (b). Longer delays indicate longer time for RF probing signal to propagate from one port to the other. Thus, both MR and CL can provide higher sensitivity in terms of frequency $f_0$ shift and $|S_{21}|_{\text{min}}$ change at 2 GHz when compared with the CPW.

The RF field intensities of the sensing areas of the three devices are also of interest. In HFSS simulation, the input power is set as 1W for each device, with average field intensity, $E_{MR}\approx2\times10^5$ V/m, $E_{CL}\approx1.3\times10^5$ V/m, and $E_{CPW}\approx6\times10^4$ V/m in the coupling gap, where there is the strongest electric field and MUT being loaded. Strong electric field will contribute to sensitivity enhancement besides slow wave effects.

Table 3.1 lists PDMS induced $S_{21}$ changes, $\Delta |S_{21}|=|S_{21}'|-|S_{21}|$ and $\Delta \angle$
$S_{21} = \angle S_{21}' - \angle S_{21}$, at a few specified frequency points. The apostrophe indicates the device with PDMS slabs. The RF interferometers will be tested in section III at these frequency points. The dimensions of the 3 PDMS slabs are 1.6× 1.8× 1.7 mm (size 1), 2.2× 4.4× 1.7 mm (size 2) and 4.1× 7.0× 1.7 mm (size 3) with an area ratio of 1:3.4:11.4. Not surprisingly, the table shows that larger PDMS MUT induces larger $\Delta|S_{21}|$ and $\Delta \angle S_{21}$ even though the ratios are not identical to the length or area (volume) ratios of the MUTs. For very small disturbance, i.e. very small MUT size or $\varepsilon_{\text{MUT}}$ close to 1 (i.e. air), a linear relationship among the ratios may be expected as long as the positions of MUTs on the device is identical each time. Table 3.1 shows that size 1 MUT on CPW causes very small changes that are within measurement accuracy limitations. As expected, MR and CL exhibit much larger $\Delta|S_{21}|$ and MR shows the strongest effects. Therefore, it is expected that MR and CL will significantly improve RF interferometer sensitivity.

**RF INTERFEROMETER PERFORMANCE ENHANCEMENTS**

The MR and CL are incorporated into the interferometer in Figure 3.1. Both simulations and measurements are conducted with PDMS as the MUT.

In simulations, a PDMS slab is loaded as indicated in Figure 3.2 (a) and (d). For the CPW, the slabs cover one of the gaps between its signal line and one ground. Then the S-parameters of these PDMS loaded/unloaded structures are obtained through HFSS simulations. The obtained S-parameters are then exported to the interferometer in Figure 3.1, which is assembled in Advanced Design System (ADS) [3.25]. With the scattering parameters of an unloaded structure, MR, or CL or CPW, the interferometer is tuned to
achieve a 540° phase difference between the reference branch and the MUT branch and a
\(~ -70 \text{ dB} \mid S_{21} \mid_{\text{min}}\) at the desired frequency. The -70 dB is our initial \(\mid S_{21} \mid_{\text{min}}\), denoted as \(\mid S_{21} \mid_{\text{ini}}\), for each set of measurement and simulation analysis later. The tuning is achieved
with the attenuator and phase shifter in the reference branch in Figure 3.1. The 540°
phase difference, instead of 180°, is consistent with the experimental setup, where the
second harmonic frequency is used. The fundamental frequency is not used because the
750MHz frequency is far from the passband of the MR. After that, the \(S\)-parameters of
the PDMS loaded structure are used to replace the \(S\)-parameters without PDMS while all
the other interferometer components are unchanged. A new \(\mid S_{21} \mid_{\text{min}}\) value is obtained at a
different frequency. Corresponding \(\Delta f\) and \(\Delta \mid S_{21} \mid_{\text{min}}\) indicate the effects of MUT, as
shown in Figure 3.4. Larger \(\Delta f\) and \(\Delta \mid S_{21} \mid_{\text{min}}\) indicate higher interferometer sensitivity.
Figure 3.5 (a) Typical measured interferometer output with (solid-dash line) and without (solid line) a size 2 PDMS slab. (b) Measured sensitivity dependence on initial $|S_{21}|_{ini}$, i.e. measured $\Delta f$ (square) and $\Delta |S_{21}|_{min}$ (triangle) at 1.925 GHz of the CPW (solid) and MR (hollow) based interferometer with size 2 PDMS slab. (c) The measured effects of PDMS slab size on sensitivity. The interferometer is initially tuned at -70 dB.
In measurements, first connect the CPW/MR/CL to the interferometer and tune the interferometer $|S_{21}|_{\text{ini}}$ to the designated initial level $|S_{21}|_{\text{ini}}$, such as -70 dB in Figure 3.5 (a). Then place the targeted PDMS slab on the coupling line gap of the MR/CL or one of the signal line-ground gaps of the CPW, as indicated in Figure 3.2. The length of the rectangular PDMS is parallel to the direction of the gap and the gap is fully covered. The measurement is verified by removing the PDMS to check if $S_{21}$ returns to $S_{21,\text{ini}}$. If it does, we can attribute the changes mainly to the MUT. The experiments are conducted at $|S_{21}|_{\text{ini}}=-60$ dB, -70 dB and -80 dB for each MUT.

Figure 3.5 (a) shows that typical measured results, which agree with simulated results in Figure 3.4 reasonably well. Similar agreements are obtained for all 3 PDMS slabs. Figure 3.4 and Figure 3.5 (a) show that the CPW and MR induce frequency changes in opposite directions and $\Delta f_{MR} \approx 2.4 \times \Delta f_{CPW}$. The ratio is smaller than the phase change ratio in Table 3.1, which can be explained by group delay differences between REF branch and MUT branch.

In the measurement frequency range, i.e. $f_0 \pm \Delta f$ in Figure 3.1, assume the phase of each branch is linear with frequency, which can be expressed as

$$P = -Tf + b$$

and $T$ is the total group delay of REF or MUT branch (Figure 3 (b)).

At $f_0$, we have

$$P_{\text{REF}} - P_{\text{MUT}} = (2n-1)\pi$$

(3.2)
After a MUT is loaded on the MUT branch, an extra phase difference $\Delta \angle S_{21}$ between MUT and REF branch is introduced. For the MUT branch, its phase is expressed by

$$P_{\text{MUT}} = -T_{\text{MUT}}f + b_{\text{MUT}} + \Delta \angle S_{21}$$

(3.3)

A new $|S_{21}|'$ occurs at $f_{0}'$, where the phase difference between the two branches is still $(2n-1)\pi$, therefore

$$\Delta f = f_0 - f_{0}' = \Delta \angle S_{21} / (T_{\text{REF}} - T_{\text{MUT}})$$

(3.4)

with $T_{\text{MUT}}$ and $T_{\text{REF}}$ the group delay of the MUT branch and the reference branch, respectively. The interferometer setup has $T_{\text{REF}}>T_{\text{CPW}}$ (a phase difference of $3\pi$, i.e. $1\frac{1}{2}$ signal period in time). Figure 3 (c) shows that $T_{\text{MR}}>T_{\text{REF}}>T_{\text{CPW}}$. The measured data in Table 3.1 show that $\Delta \angle S_{21,\text{MR}}$ and $\Delta \angle S_{21,\text{CPW}}$ are both negative. Thus, $\Delta f$ for MR and CPW have opposite signs. And the $\Delta f$ change ratio is reduced because of the difference between $T_{\text{REF}}$ and $T_{\text{MUT}}$. Nevertheless, these issues can be addressed by using an identical MR or CL in the REF branch.

Figure 3.5 (a) shows that MR helps sharpen $|S_{21}|$ so the effective quality factor is $Q_{\text{MR}}\approx 16000$, which is much higher than $Q_{\text{CPW}}\approx 3500$ when $|S_{21}|_{\text{ini}}$ is $\sim -70$ dB. Thus, better frequency resolution can be achieved with MR. After the introduction of PDMS slab, however, $Q_{\text{MR}}<Q_{\text{CPW}}$ because of much stronger PDMS effects. It is also shown that $\Delta |S_{21}|_{\text{min,MR}}=36.0$ dB while $\Delta |S_{21}|_{\text{min,CPW}}=10.7$ dB
The change of $|S_{21}|$ at $f_0$, denoted as $\Delta |S_{21}|_{f_0}$, can also be used to sense MUT. As is shown in Figure 3.5 (a), $\Delta |S_{21}|_{f_0}$ is much larger than $\Delta |S_{21}|_{\text{min}}$. Therefore, it is a much more sensitive sensing indicator. However, in the following analysis we continue to use $\Delta f$ and $\Delta |S_{21}|_{\text{min}}$ for sensitivity discussion.

Figure 3.5 (b) shows the effects of $|S_{21}|_{\text{ini}}$ on interferometer sensitivity at 1.925 GHz with size 2 PDMS slab. Compared with CPW structure, MR induces ~ 4 times higher $\Delta f$ and up to 40 dB larger $\Delta |S_{21}|_{\text{min}}$. It also shows that for both MR and CPW structures, $\Delta f$ does not depend on $|S_{21}|_{\text{ini}}$ since $\Delta f$ is associated with $\Delta \angle S_{21}$, which does not depend on $|S_{21}|_{\text{ini}}$. As long as the MUT introduced phase change is the same, $\Delta f$ should be the same. Yet $\Delta |S_{21}|_{\text{min}}$ depends on $|S_{21}|_{\text{ini}}$ nonlinearly. Lower $|S_{21}|_{\text{ini}}$ results in larger $\Delta |S_{21}|_{\text{min}}$. Conceptually, when $|S_{21}|_{\text{ini}}$ is low and MUT effects are strong, $|S_{21}|_{\text{min}}$ is expected to reach the same value for all $|S_{21}|_{\text{min}}$ when MUT is applied. Thus, we expect to observe $\Delta |S_{21}|_{\text{min}}$ differences equal to $|S_{21}|_{\text{ini}}$ difference. The variations in Figure 3.5 (b)
are probably caused by the repeatability issue when manually placing PDMS samples. Another possible reason is that the attenuator is tuned differently for different $|S_{21}|_{ini}$. So the operating frequencies differ slightly due to inevitable phase differences induced by the attenuators. As a result, frequency dependent attenuation effects may also occur in the observed $\Delta|S_{21}|_{min}$.

Figure 3.5 (c) shows the effects of PDMS slab size on interferometer sensitivity when $|S_{21}|_{ini}$ is ~ -70 dB at 1.925 GHz. It shows that $\Delta f$ increases with MUT size. And when we compare the $\Delta f$ and $\Delta \angle S_{21}$ of the three PDMS MUTs in Table 3.1 Broadband measurements of $S_{21}$ changes (magnitude and phase) induced by PDMS slabs at the targeted frequencies., the ratios of are slightly different since $\Delta \angle S_{21,1} : \Delta \angle S_{21,2} : \Delta \angle S_{21,3} = 1: 2.8 : 6.2$, while $\Delta f_1 : \Delta f_2 : \Delta f_3 = 1 : 3.6 : 5.8$. The discrepancy may come from MUT positioning variations. In broadband measurements, the accuracy is also limited.

For MR and CPW, larger size PDMS will produce larger $\Delta|S_{21}|_{min}$ even though size 2 and size 3 MUT of MR show similar values.

Figure 3.6 presents typical simulated interferometer results, which are similar to measured curves. CL structures yields larger frequency shift and magnitude change. It shows that (i) $\Delta f_{CL}$=4.0 $\times$ $\Delta f_{CPW}$, (ii) $Q_{CL}$=5500, $Q_{CPW}$=3500, and (iii) $\Delta|S_{21}|_{min, CL}$=17.2 dB, $\Delta|S_{21}|_{min, CPW}$=6.2 dB. For size 2 PDMS, simulations show a $\Delta f$ = 20 MHz, which is larger than $\Delta f$ = 11 MHz observed in experiment. A possible reason for the discrepancy is the frequency dependent attenuator and phase shifter characteristics as well as extra phase delay that are not included in simulations.
Figure 3.7 (a) Measured Δf (square marked) and Δ|S_{21}|_{min} (triangle marked) at 2.2 GHz of the CPW (black) and the CL (hollow) of small size MUT (b) Measured Δf and Δ|S_{21}|_{min} of three sized of MUT at -70 dB (the same marker as (a)) at 2.2 GHz (1-small, 2-middle, 3-large) (c) Measured Δf or Δ|S_{21}|_{min} of the size 1 MUT at -70 dB at each frequency point (the same marker as (a)).
Figure 3.7 (a) shows the effects of $|S_{21}|_{ini}$ on interferometer sensitivity at 2.2 GHz with size 2 PDMS slab. It shows that $\Delta f_{CL} \approx 3-4 \times \Delta f_{CPW}$ with $\sim 30$ dB better $\Delta |S_{21}|_{min}$; $\Delta f$ does not depend on $|S_{21}|_{ini}$ while $\Delta |S_{21}|_{min}$ does, similar to those results of MR.

The process of magnitude changes can be analyzed as the following. Assume the $|S_{21}|$ of MUT and REF branch does not change in the vicinity of operating frequency $f_0$. The assumption is reasonable for CL and CPW as the zoomed in frequency band is narrow, i.e. 60MHz in Figure 3.6. For MR, its $|S_{21}|$ change could be as large as the change induced by the MUT, therefore requiring further analysis in the future.

The magnitude of the two branches are denoted as $A_1(\omega)$ and $A_2(\omega)$. Before the MUT is added on the device,

$$|S_{21}|_{ini} = \frac{|A_1(\omega_i) - A_2(\omega_i)|}{2} \quad (3.5)$$

After the MUT is put on the, $|\Delta A_1|$ is the magnitude change induced by the MUT in the sensing branch, while the reference branch remains the same. Therefore,

$$|S_{21}|_{min,MUT} = \frac{|A_1'(\omega_2) - A_2(\omega_2)|}{2}$$

$$= \frac{|A_1'(\omega_2) - A_1(\omega_i) + A_1(\omega_i) - A_2(\omega_2)|}{2} \quad (3.6)$$

The changes in decibels is

$$\Delta |S_{21}|_{min} = 20\log\left(\frac{|S_{21}|_{min,MUT}}{|S_{21}|_{ini}}\right) \quad (3.7)$$

It indicates that both MUT and $|S_{21}|_{ini}$ will contribute to $\Delta |S_{21}|_{min}$.

Figure 3.7 (b) shows the effects of PDMS slab size on interferometer sensitivity when $|S_{21}|_{ini}$ is $\sim -70$ dB at 2 GHz. Frequency shift $\Delta f$ increases with MUT size. Direct
broadband measurements give $\Delta S_{21,1} : \Delta S_{21,2} : \Delta S_{21,3} = 1.0 : 2.0 : 4.5$, while $\Delta f_1 : \Delta f_2 : \Delta f_3 = 1.0 : 2.6 : 5.0$ for CL. Similar results are obtained at other frequency points.

Figure 3.7 (c) shows that the performance of CL based interferometer does not have obvious frequency-dependent characteristics even though the $\Delta f$ of CPW increases with $f$ (which is expected). The observed larger $\Delta|S_{21}|_{\text{min}}$ and $\Delta f$ at 2.2 GHz for CL interferometer may be partly due to the filtering characteristics in Figure 3.4. Nevertheless, CL yields much higher sensitivity at all frequency points.

Compare the results of MR and CL in Figure 3.5 and Figure 3.7, it shows that MR based interferometer has higher sensitivity and higher resolution ($Q$ values), which qualitatively agree with the group delay prediction in Figure 3 (c). The discussions about MR group delay in sub-section A above and MR higher insertion loss all contribute to the observed interferometer sensitivity results.

TRANSMISSION ESR OPERATION WITH THE MICRO-COIL MR

So far, we have proved that substituting the CPW with a resonant structure can improve the detection sensitivity. However, in order to detect magnetic property of the MUT, the sensing zone should be at the center of the semi-ring where the current reaches its maximum at resonance and it generates strong magnetic field [3.23].

The resonator is re-designed to working at higher frequency where ESR signal is stronger [3.26] and the dimension of the resonant can be further minimized due to shorter wave length, thus increasing the filling factor which is helpful to improve the sensitivity.
The MR is shown in Figure 3.8 (a) is designed to investigate the performance of the system Chapter I. Standard microfabrication techniques are used to build the MR and MML on a 1mm thick silica substrate ($\varepsilon_r=3.8$) with 2 $\mu$m gold films. The MR has an imbedded planar micro-coil with $R_1 = 0.5$ mm, $R_2 = 0.7$ mm, $G = 0.06$ mm, $W_1 = 0.4$ mm,
and \( W_2 = 2.88 \text{ mm} \) (Figure 3.8 (b)). The RF magnetic field distribution of the MR is similar to that in [3.22]. In ESR measurement, MUT DPPH is placed in the micro-coil. Figure 3.8 (c) shows its equivalent circuit. Inductance \( L \) and resistance \( R \) include DPPH contributions. Figure 3.8 (d) shows that the resonance is centered at \( \sim 7.6 \text{ GHz} \) with an unloaded quality factor \( Q_0 = f_0/f_{3\text{dB}} \approx 19 \), where \( f_0 \) is the resonant frequency and \( f_{3\text{dB}} \) the 3-dB.

The MR in Figure 3.8 is incorporated into the ESR system in Chapter I. At \( \sim 7.6 \text{ GHz} \), the interferometer is working at its 3\textsuperscript{rd} harmonic frequency, determined by the electrical length difference between the two branches. To overcome the difficulty in weighing sub-microgram DPPH for test, 400 \( \mu \text{g} \) DPPH powder is first dissolved into 200 \( \mu \text{l} \) toluene. Then 0.1 \( \mu \text{l} \) solution is dropped on to the micro-coil. The estimated DPPH is 0.2 \( \mu \text{g} \) \((2.8 \times 10^{14} \text{ spins})\). The microwave power from the VNA is 6 dBm. The initial \(|S_{21}|_{\text{min}}\) is tuned between -95 dB and -105 dB. Figure 3.9 (a) shows some typical VNA readings. Figure 3.9 (a), (b) and (c) show the magnitude change \( \Delta |S_{21}|_{\text{min}} = |S_{21}|_{\text{min}} - |S_{21}|_{\text{min,ini}} \) and frequency shift \( \Delta f = f_0 - f_{0,\text{ini}} \) at different \( B_{\text{ext}} \), where the \( |S_{21}|_{\text{min,ini}} \) and \( f_{0,\text{ini}} \) are the initial \(|S_{21}|_{\text{min}}\) and \( f_0 \), respectively. It shows that \( \Delta |S_{21}|_{\text{min}} \) vs. \( B_{\text{ext}} \) follows \( \mu'' \), while frequency shift \( \Delta f \) resembles the dispersion curve of \( \mu' \).

However, \( \Delta |S_{21}|_{\text{min}} \) is not symmetric and \( |\Delta f_{\text{max}}| \neq |\Delta f_{\text{min}}| \). There are several possible reasons. First, the resonator responds to positive \( \chi' \) and negative \( \chi' \) differently. This can be shown through an HFSS simulation on \( \chi' \) induced phase shift \( \Delta \phi \). For a 2mm\( \times \)2mm\( \times \)0.01mm MUT slab covering the half-ring in Figure 3.8 (a), a \( \chi' = 0.004 \) induces a \( \Delta \phi = 0.1^\circ \) while \( \chi' = -0.004 \) induces \( \Delta \phi = -0.4^\circ \). Thus, \( |\Delta f_{\text{max}}| \) is not equal to
Δ|f_{min}| in Figure 3.9 (c). Secondly, μ' causes dispersion and re-centers the working
frequency of the resonator. Thus, it causes a $\Delta |S_{21}|_{\text{MR}}$ at the measurement frequency, which induces an asymmetric $\Delta |S_{21}|_{\text{min}}$. Such phenomenon is mitigated when we substitute the resonator with the MML below, where the phase change is more linear to $\chi'$ and dispersion is less likely to produce a $\Delta |S_{21}|_{\text{MML}}$.

The measurement in Figure 3.9 (b) gives $N_s = 2.52 \times 10^{-6}$ and an $\text{SNR} = 121$ with 0.2 µg DPPH. The sensitivity is higher than the MML measurement measuring 6 µg and obtaining $\text{SNR} = 194$.

**REFLECTION ESR OPERATION WITH THE MICRO-COIL MR**

Aside from transmission operation, in which $S_{21}$ is measured, the interferometer can also operate in a reflective way, i.e. measuring $S_{11}$. Therefore, the interferometer is adapted to accommodate to such measurement as the Figure 3.10 shows. Different from
the transmission interferometer, the probing wave and reference wave are reflected and combined again at the input power divider with \((2n+1)\pi\) phase difference. To realize such reflection, at the reference end, a short or an open load can be used, while at the measurement branch we need a reflective resonator, like those used in [3.27].

The resonator is built on a high dielectric substrate of 36. The purpose of the using such substrate is to decrease the size of the sensing zone, since the guided wavelength on a piece of microstrip line equals

\[
\lambda = \frac{3 \times 10^8 \text{ m/s}}{f \sqrt{\varepsilon_{\text{eff}}}}
\]  

(3.8)

which is inverse to the square root of the effective permittivity [3.23]. Compared with quartz substrate, with the same thickness of 1 mm, the guided wavelength is minimized.
by around three times on the high permittivity substrate. If we measure a material of the same size, the filling factor $\eta = \frac{V_{MUT}}{V_R}$ will increase. As it is derived in [3.18], the ESR signal equals

$$V_s = (1/2) \chi'' \eta Q_L Z_0 P$$

(3.9)

Figure 3.11 (a) is the center part of the resonator. The actual chip size is 25.4 mm×12.7 mm with 50 Ω line extended to each end. The probing wave will be coupled to the resonant part and reflected to the input port. The total length of the resonator is $\lambda$ to ensure that the sensing zone is about $\lambda/2$ long and the current at the sensing zone has the same direction, besides the $\lambda/4$ coupling section. Figure 3.11 (b) is the field distribution of the $z$ component of the magnetic field at resonance frequency. It can be observed that at the center of the sensing zone.

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Figure 3.12 Simulations (dash line) and measurements (solid line) of $|S_{11}|$ (blue) and $|S_{21}|$ (red) of the resonator.
the field is uniform while at the gap between the transmission lines the field is cancelled to be null due to the current has the same direction at each side. Figure 3.11 (c) is shows the side view of the magnetic field along the center line annotated in the layout. The magnetic field decreases to \( h = 0.3 \) mm from the surface of the device.
The dashed lines in Figure 3.12 are the simulated $S$-parameters. Due to the resonator’s length in $\lambda$, we can observe a lower harmonic along with the resonance $f_0 = 8.64$ GHz. However, in the measurement, the pin size of the connector is 0.2 mm which is wider than the 50 $\Omega$ line and misalignment makes the mismatch even worse. Consequently, the resonance is overwhelmed by the reflection. Probably a microwave probe is better than the conventional SMA connector in this case. By reading the $|S_{21}|$, its minimum point corresponds to the resonance which agrees well with the simulation and it is the working frequency of the spectroscopy.

A single TEMPOL crystal is placed on the sensing zone of the resonator as shown in Figure 3.10 (d). Due to the high substrate permittivity, the resonance frequency is insensitive to the crystal. TEMPOL is another standard free radical commonly used in ESR measurement with $g = 2.006$. The measurement procedures are similar to those in the transmission measurement: first the balance of the interferometer is tuned and then the magnetic field is swept. However, the $S_{11}$s instead of $S_{21}$s are recorded.

Figure 3.13 (a) is several typical measurements. We can observe the $|S_{11}|$ changes with the external magnetic field. And Figure 3.13 (b) is the absorption and the dispersion is recorded in Figure 3.13 (c). Following previous definition $\text{SNR} = 120$.

Compared with previous measurements, the sensitivity improvement is not that obvious even if the sensing zone is minimized. Several reasons may account for this result. The first is the performance of the resonator. Much less microwave is coupled to the resonator due to the connection problem by the connectors. Also, the low $Q$ value of the resonator caused by over coupling lower the sensitivity. Furthermore, the field
cancellation in the line gap decreases the effective sensing area making less material is detected. A field integration is conducted to calculate the \( B_z \) over the sensing zone. Because of the such cancellation, compared with sensing same area on the previous transmission resonator, the field integration only increases by 1.5 times. Last but not the least, the wave travels twice through the attenuators and the phase shifter, the thermal and mechanical vibration noises are doubled.

DISCUSSIONS AND CONCLUSIONS

In this chapter, first, PDMS slabs, i.e. dielectric materials, are used in the above simulation and experiments to demonstrate that MR and CL can significantly improve RF interferometer sensitivities. Enhanced RF electric fields are exploited for the process. It can be shown that CL and MR can also be exploited for high sensitivity magnetic material detection. For example, HFSS and ADS simulations show that a magnetic material (0.2 × 0.2 × 0.2 mm) with \( \mu' = 2.4 \) and magnetic loss tangent of 0.001 will induce a \( \Delta f = 13.0 \) MHz and \( \Delta |S_{21}|_{min} = 25.3 \) dB when loaded in an MR based interferometer. The permeability is close to that of some ferrite composite, and the material is placed on the edge of the split-ring, shown in Figure 3.2 (a) and similar to that in [3.22]. The interferometer is tuned at 2 GHz with a \( |S_{21}|_{min} = -70 \) dB. When the same MUT is put in the gap between the signal line and the ground of the CPW, only \( \Delta f = 0.2 \) MHz and \( \Delta |S_{21}|_{min} = 5.2 \) dB are induced.

The large size MR, CL and CPW devices used in this work enabled convenient experimental processes, including manual positioning of large size PDMS slabs. But the
processes cause relatively large repeatability issues as well as compromised sensitivity comparisons since the effective amount of PDMS that is interacting with RF E-fields is not necessarily the same. Furthermore, the use of large PDMS slabs causes large \( \Delta f \) and \( \Delta |S_{21}|_{\text{min}} \), which impede detailed study processes, especially when \( \Delta |S_{21}|_{\text{ini}} \) is very low. Frequency and magnitude dependent interferometer properties are difficult to include in data analysis. However, when micro-nanofabrication techniques are used to build these devices and position the MUT, the issues can be addressed. Furthermore, HFSS simulations of a microfluidic channel whose cross view area of 50 \( \mu \text{m} \times 80 \mu \text{m} \) filled with DI-water vertically passing the gap of MR and CL indicate that the MR and CL still function well, which provides the potential for the device to measure liquid. After all, small amount of liquid does not produce significant impact on the broadband performance of the device, even if it is highly dispersive in RF frequencies.

To sum up, the simulations and measurements show that MR and CL slow down RF wave propagations and enhance local RF fields, compared with the performance of a reference CPW. As a result, interferometer sensitivities are significantly enhanced, as demonstrated by measured results at different frequency points with different PDMS slabs and at different initial interferometer operation levels. Equivalently, smaller detection dynamic range is needed to measure a given MUT sample. More work is needed to quantify the processes and obtain material properties.

Secondly, the method verified by the dielectric detection is then applied to the ESR measurement. The transmission and reflection resonators substitute the microstrip meandered line. As a result, the sensitivity is significantly enhanced: 0.2 \( \mu \text{g} \) (2.8\( \times \)10\(^{14} \))
spins) DPPH (2,2-diphenyl-1-picrylhydrazyl) sample is measured at a signal-to-noise ratio (SNR) of ~ 121. And the sensitivity can be even better than our current results because, the resonator in Figure 3.8 is ~ 5 times larger in diameter (D) than the resonator in [3.22], which could reduce ESR sensitivity by a factor of ~ 4 due to the $1/D^{0.8-1}$ scaling [3.28]. Secondly, the resonator in [3.22] works at 14 GHz and has a $Q$-value of 37 while the resonator in Figure 3.8 (a) is at ~ 7.6 GHz with a $Q$ of 19. Higher frequency [3.29] and higher $Q$ [3.27] will result in higher sensitivities proportionally. Thirdly, the magnetic fields on some of the sensing zone of the reflective resonator are zero. Consequently, the effective volume of the MUT interacting with microwave is much less than the actual size of the crystal.

REFERENCES


CHAPTER FOUR

FIELD MODULATED ESR WITH THE RF INTERFEROMETER

INTRODUCTION

So far, we have demonstrated that the ESR spectrometer by the RF interferometer can help the VNA can successfully detect the ESR signals by removing the unwanted probing wave. However, the RF interferometer is a passive system, which is not able to improve the signal to noise ratio by itself. Then how it makes it possible to detect such small ESR signal is a question should be answered, which is the purpose of this chapter.

In previous measurements, we took the VNA as a highly integrated ‘black box’ and mainly focused on the outputs instead of the detection process. So as to answer the question above, we need open the ‘black box’ and reveal the signal process to fully understand the role of the RF interferometer. Therefore, we decide to seek to the conventional lock-in detection with field modulation, which is essentially a similar detection method to the VNA. Different from the integrated VNA system, in the lock-in detection, some components can be pulled out to give us a straightforward understanding of how the receiver in the VNA works. Also, it is also of great interest to explore if the RF interferometer can bring benefits to the lock-in ESR detection.

Even if the lock-in detection has some issues mentioned before, it is a powerful phase-sensitive technique that can detect small ESR signals from overwhelming noise background [4.1-3]. The general idea of this technique is to transfer the interested signal to the side band of the carrier wave and the lock-in amplifier provides a reference frequency which can filter out the ESR signal at the side band after a mixer. Noises at
other frequencies, like the environmental noise will not interfere with the detection results. Aside from ESR detection, this technique is also used to build other bio-sensors where the signals generated by cells are also too small to be detected by a regular detection instrument [4.4].

In this chapter, first the working principles of lock-in detection together with the case with an RF interferometer are introduced. After the systematic theoretical analysis, the conventional ESR is built accordingly with its operation parameters optimized. Then the RF interferometer is incorporated into the system, by which the ESR is measured. Finally, measurement results are compared and discussed.

**LOCK-IN DETECTION PRINCIPLES**

The principles of lock-in detection and its applications are introduced in [4.1-3]. The operation relies on the coherence to the reference signal generated by the lock-in amplifier \( V_R(t) = \sin(\Omega t) \). If we have an input signal \( V(t) = V_0 \sin(\omega t+\varphi) \), at the lock-in detector the signal will have a product with the reference signal resulting in the sum and the difference of frequencies:

\[
V_R(t)V(t) = \frac{V_0}{2} \left\{ \cos[(\omega-\Omega)t+\varphi] - \cos[(\omega+\Omega)t+\varphi] \right\}
\]  

(4.1)

If the reference signal and the input signal have the same frequency, the output signal will contain a DC bias and a high frequency AC component, and the DC bias will be kept by a low-pass filter. However, if they do not agree, the input signal will be rejected. Therefore, the lock in amplifier only reads signal at the same frequency with the reference or those tolerated by the passband of the lowpass filter.
Moreover, the phase $\phi$ will impact the magnitude of the detected signal. In ESR process, the dispersion will change the input signal phase, which may distort the signal. Modern lock-in amplifier can solve the issue easily by adding a second phase sensitive detector (PSD) and adding an extra 90° phase [4.5]. Now we have two signals: $X =$

Figure 4.1 The output ESR signal (yellow) of excited by a modulated external magnetic field (red) on a piece of linearized absorption curve (blue).

Figure 4.2 The absorption of ESR and its first derivative detected by the lock in amplifier.
$V \cos(\varphi)$ and $Y = V \sin(\varphi)$ and the magnitude can be easily computed by $V^2 = X^2 + Y^2$ and the phase is $\varphi = \tan^{-1}(Y/X)$.

After understanding the basic working principle of the lock-in detection, we can consider its application to ESR detection. To generate the ESR signal at the same frequency with the reference signal $\omega_R$, aside from the DC magnetic field, a modulated AC magnetic fields at $\omega_M = \omega_R$ is also applied, which is

$$B(t) = B_1 + B_M \cos(\omega_M t) \quad (4.2)$$

The response of the material to the resonator is a voltage signal dependent on the total magnetic field $V(B)$. In practice, we set $B_M << B_1$ and the Taylor’s expansion of $V(t)$ can approximate $V(B)$ at $B_1$:

$$V(t) = V(B_1) + \frac{dV}{dB} \bigg|_{B_1} B_M \cos(\omega_M t) \quad (4.3)$$

Lock-in amplifier can pick up the second term which has the same frequency with the its reference.

Figure 4.1 gives a visualization of the working principles described above while the absorption of the ESR and its first derivative by the lock-in detection are shown in Figure 4.2.
LOCK-IN ESR DETECTION SYSTEM

In this experiment, we first build a conventional lock-in ESR system and then incorporate the RF interferometer into it. The schematic of the lock-in ESR system is shown in Figure 4.3 which is similar to those in [4.6, 7]. The major difference is that they use a one-port reflective resonator together with a circulator while in our experiment a two-port transmission resonator is used.

The RF source generates the probing wave which is divided equally by the power divider. One of them works as the reference signal, that is, local oscillating signal (LO) into the mixer. After an optional attenuator, the remaining microwave signal interacts with the ESR sample through the transmission resonator. There is a DC permanent magnet together with the home-made modulation coil provides the required magnetic fields. The bipolar amplifier amplifies reference signal and drives the modulation coil. The 3-dB attenuator before the mixer avoids the reflections. These reflections are caused by the mismatch of the mixer whose VSWR is ~2.5 at the RF input port and they may severely distort the signal. The T-junction with 50 Ω termination in parallel with the LIA input impedance (1 MΩ) and therefore, it matches the LIA to the 50 Ω system. This T-junction also protects the LIA from being damaged by high power signal because most of the power will be diverted to and absorbed by the 50 Ω termination. The models of the components are specified in the caption of Figure 4.3.

Figure 4.4 The signal ESR detection signal flow of the conventional method. \( \omega_0 \) is the microwave frequency and the \( \omega_m \) is the modulation frequency.
Based on the knowledge in the lock-in detection principles and the system schematic, we can build a signal flow with mathematic expressions and plots for the ESR detection in Figure 4.4. In the sensing part, due to the oscillation of the magnetic field, the ESR absorption to the microwave also oscillates with the AC field. As a result, the probing wave experiences a magnitude modulation by the ESR. At the mixer, a product is made so that the difference frequency agrees with the reference. This signal will be

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received by the LIA and the IQ demodulation within can generate both the DC magnitude and phase ESR signal.

With the lock-in ESR system built, incorporating the RF interferometer into the system is the second step. The newly built system shown in Figure 4.5 is similar to the original system, except the resonator is connected into the RF interferometer first and the interferometer replaces the resonator in its original system.

With the RF interferometer canceling out the unwanted probing wave at the output, the signal flow chart is also modified in Figure 4.6. Obviously, unlike that in Figure 4.4, with the interferometer, the input of the mixer only contains the modulated
ESR signal. This setup provides significant dynamic range to the detection system: it is entirely feasible to add an amplifier to increase the intensity of the signal without causing non-linearity issue to the mixer.

Figure 4.7 The resonator and its simulated and measured results. (a) The layout of the resonator, $R_1 = 0.5$ mm, $R_2 = 0.7$ mm, $L_1 = 0.4$ mm, $L_2 = 18.96$ mm, $g = 0.02$ mm and $W = 2.7$ mm. (b) The simulation and measurement results.
Finally, the resonator in this experiment is introduced here. Drawn in Figure 4.7 (a), it is very similar to the resonator we used before, but with the physical length increased, the resonator resonates at 3.079 GHz by measurement, which is the working of the ESR system hereafter. The reason for the lower frequency operation is to accommodate to the components available for us.

Admittedly, higher frequency can help the sensitivity. Nevertheless, for the functional exploration and comparison purpose, a lower frequency resonator is sufficient.

The simulation and measurements results of the resonator are shown in Figure 4.7 (b). Only a small frequency shift is observed which is probably caused by the discrepancy between the real metal thickness and simulated one, resulting in a different coupling capacitance.
LOCK-IN ESR EXPERIMENTS WITH THE RF INTERFEROMETER

Before incorporating the RF interferometer into the lock-in detection system, the measurements with the lock-in system only are performed in which the non-linear effects will be shown, and the results will be compared with those when the RF interferometer is added. Figure 4.8 shows the lock-in detection system with the RF interferometer. The free radicals used in this experiment is about 1 µg TEMPOL crystal (g = 2.006 [4.8]). The source generates microwave of 16 dBm. And the lock-in amplifier is set as: time constant 10 ms, filter slope 24 dB/oct and ENBW = 7.1 Hz.

In the lock-in ESR experiment, the -10-dB attenuator is first connected to reduce the input power of the resonator and then it is removed. The experiment results are shown in Figure 4.9 and the SNRs are computed as the peak-to-peak value of the signal to the RMS value of the noise. With the attenuator, a $SNR = 231$. Without the attenuator, the input power to the mixer is much higher.

Consequently, the non-linear effects caused by the mixer distort the ESR signal and with a $SNR$ to 412. Considering that the 1-dB compression point of the mixer is 9 dBm, it is unsurprising to observe such non-linear effects.

After this experiment, the resonator is disconnected and reconnected into the RF interferometer. The RF interferometer is connected to the VNA and tuned to ~60dB and then put into the measurement branch of the lock-in detection system. This cancellation provides a significant dynamic range and enables the amplifier with 22 dB gain connected after the interferometer.
Figure 4.1 shows the measurement with and without the amplifier. The measurement does not require the -10 dB attenuator; as a result, the input power is much higher without causing any nonlinear effects. The obtained $SNR = 175$ which is lower

Figure 4.9 The measurement by the lock-in ESR detection system with and without the attenuator at the input end.

Figure 4.10 The measurement by the lock-in ESR detection system and the RF interferometer with and without the amplifier at the output end before the mixer.

Figure 4.10 shows the measurement with and without the amplifier. The measurement does not require the -10-dB attenuator; as a result, the input power is much higher without causing any nonlinear effects. The obtained $SNR = 175$ which is lower
than what we obtained without the RF interferometer. The reason might be the lack of the carrier signal making the signal vulnerable to the noise of the detector. However, if we connect the amplifier, even if the output power is still only—30 dBm, which is far from
the saturation range, the obtained signal to noise ratio increases to 1450 due to the 22-dB gain of the amplifier.

Aside from avoiding non-linear effects, we can also look at the improvements by the interferometer on the phase detection. Based on the LIA working principles and signal flow in Figure 4.4 and Figure 4.6, if there is a phase change of the resonator caused by the ESR process, the LIA can detect such signal. And our previous experiments with the VNA verify that an extra phase change does occur in the ESR process. Figure 4.11 shows the phase \( \theta \) measured by the lock-in detection and with the RF interferometer. It can be observed that with the RF interferometer, the phase change is clearly presented which follows the dispersion of the ESR; however, without the RF

![Figure 4.13](image-url)

Figure 4.13 Vector illustration of the RF interferometer output phase \( \theta' \) is larger than the phase \( \theta \) generated by the resonator in the ESR process.

![Figure 4.14](image-url)

Figure 4.14 Noise figure of the mixer-amplifier and amplifier-mixer cascade.
interferometer we can only read the 180° change caused by the polarity of the first derivative of the ESR absorption curve.

The reason for this phase reading is associated with the phases of the resonator and the interferometer. Figure 4.12 shows the comparison: with the interferometer, the phase changing rate is much higher than the resonator itself and the interferometer is more sensitive to phase change. The sensitivity improvement by a resonator or seconds stage interferometer is verified by the previous work in Chapter II and the publication of [4.9, 10].

In addition to that, the vector illustration in Figure 4.12 can also help to explain. At the initial state, the RF interferometer is well tuned and MUT branch and REF branch has a 180° difference. If the ESR process introduces a phase $\theta$, the phase of the summation of the two vectors $\theta'$ is larger than $\theta$ if the magnitude $|M| \approx |R|$ in that,

$$\theta' = \arctan \left( \frac{\sin \theta}{\frac{R}{M} \cos \theta} \right)$$  \hspace{1cm} (4.4)

Finally, let’s compare another possibility of adding an amplifier after the mixer to increase the input signal. The model can be simplified into the two scenarios shown in Figure 4.14. The cascade noise figure $F$ can be computed as [4.11],

$$F = F_1 + \frac{F_2 - 1}{G_i}$$  \hspace{1cm} (4.5)

By putting the numbers into the equation, in case (a), the $F = 7.4$ dB while in case (b), the $F = 1.46$ dB. Therefore, due to the conversion loss, if we put the amplifier after the mixer, the noise figure will increase by 6 dB. This comparison shows that our method is better.
VNA DETECTION WITH THE RF INTERFEROMETER

After understanding how the RF interferometer increases the dynamic range of the conventional lock-in detection technique, let’s revisit the detection process with the VNA. Figure 4.15 is a simplified VNA receiver functional schematic [4.12, 13]. Compared with the lock-in detection, the difference is the ESR signal is at the same frequency with the carrier wave. Otherwise, the principles are similar, both involving frequency down-conversion and digital processing.

The first contribution by the RF interferometer is at the mixing part. The RF interferometer can significantly reduce the RF power, as a result, avoiding the non-linear effect at the mixer. The second contribution is to the ADC process. The number of bits of the ADC is limited and the resulting resolution determines its accuracy. Imaging the unwanted probing wave is not removed, most of digits will be used to characterize these signals and the small ESR signal can be overwhelmed by the ADC errors. However, if only the ESR signal is kept, with proper tuning of the pre-amplifier, all of the digits of the ADC will be used to translate the ESR signal, resulting in a much higher accuracy. Based

Figure 4.15 Schematic of the VNA’s receiver.
on these two contributions, the RF interferometer can abstract the minor ESR signal out can let the VNA focus on the ESR signal only.

DISCUSSIONS AND CONCLUSIONS

In this chapter, first the lock-in detection principles with and without an RF interferometer are introduced. The signal flow indicates that the RF interferometer is able to remove the unwanted probing wave and keep the ESR signal only. This method can significantly increase the dynamic range of the system and avoids the nonlinear effects at the mixer.

After the theory introduction, we use two experiments to demonstrate the idea. One is a conventional lock-in ESR detection and the other is the modified one by incorporating the RF interferometer. In the first experiment, the non-linear effect can be observed due the high voltage input the RF port. However, if we add the RF interferometer, we can freely add an amplifier without causing any non-linear problem. And the amplified signal can overcome the noise at the receiver of the LIA. As a result, the SNR is significantly increased.

However, the amplifier we use will decrease the SNR of the signal itself due to its noise figure. And there is another option avoid the noise of the receiver by adding a pre-amplifier before the LIA. Therefore, further work should be made to investigate this issue and compare the two scenarios by experiment.

Moreover, the RF interferometer will change the absorption signal since the extra phase cause a frequency shift. And if we are reading the data at one frequency, such
frequency shift may also introduce a magnitude change. In our measurement, due to the little amount of material, such frequency shift is nearly not discernable. But if the frequency shift is large enough, its effect on the magnitude change should be taken into consideration. And in this case, the signal we detect is not the magnitude information anymore but a mixture of the magnitude and phase change.

In conclusion, we have studied the effects of the RF interferometer on the ESR detection. It does help the detection process by avoiding the non-linear effect and increase the accuracy of ADC. By adding an amplifier after the RF interferometer, the signal is increased to overcome the noise at the detector. As a result, the SNR is significantly increased.

REFERENCES


CHAPTER V
HIGH FREQUENCY SENSOR DEVELOPMENT

INTRODUCTION

Previously, we have demonstrated the interferometer-based sensors working at lower than 15 GHz where the performance of the planar circuits is satisfactory. However, at higher frequency range, especially at Terahertz (THz), the insertion loss of the planar circuits (microstrip lines or coplanar waveguide) can be problematic due to conduction loss and the unwanted waveguide mode propagating in the substrate between the conductors. Therefore, waveguide is a prevalent transmission line at mm wave range [5.2].

Even if the performance of waveguides is better than the planar circuits at mm wave range, using waveguides as the sensing device can be problematic, since its dimension is dependent on the working frequency and at lower frequencies, the waveguides can be much bulkier than the planar circuits whose electric or magnetic field energy is less concentrated than that in the planar circuits. The purpose of this chapter is to demonstrate the idea of concentrating the electric fields in the waveguide by using two structures, a ridged-waveguide and a tapered waveguide.

With frequency going even higher into THz range, developing sensors is of great popularity recently [5.3-7]. The working principles are similar to low frequency microwave sensors, the refractive indices caused by the dielectric properties of the MUT can be extracted from the transmitted THz spectra [5.8]. Beside dielectric properties detection, THz ESR is a very important domain for the resonance spectroscopy in
magnetic fields, which has several advantages over conventional ESR methods [5.9], since it can reveal rich information content in chemistry, biology, and materials science [5.10]. For example, $g$ value resolution can be greatly improved in the extended frequency-magnetic field region, and an ESR transition across the zero-field splitting becomes possible with higher-energy electromagnetic waves [5.9]. Therefore, designing a THz RF interferometer-based sensor for dielectric material characterization and ESR spectroscopy with high sensitivity is of great interest.

This chapter is arranged as the following. First a ridged waveguide is designed and tested. And the ridged-waveguide is incorporated into the RF interferometer to test small amount of aqueous mixture to prove its functionality. The second proposed structure is a tapered waveguide. The vertical dimension of the waveguide is reduced to enhance the electric field. The MUT will affect its cutoff frequency. Finally, the integrated THz interferometer working at ~298 GHz is presented. Simulations show that by properly positioning the MUT according to the field distribution, the integrated spectrometer is able to detect both the dielectric and magnetic properties change of the MUT, which provides the possibility of testing ESR signal at THz.

RIDGED-WAVEGUIDE SENSOR

Compared with standard waveguide with the same internal dimensions, ridged-waveguide has a lower cutoff frequency and characteristic impedance [5.11, 5.12]. Figure 5.1 (a) and (b) shows its cross view and equivalent circuit [5.1]. Due to the proximity of the two ridges, the equivalent capacitance is larger than a conventional waveguide and
the equations in [5.1] can find the values of the equivalent capacitance and inductance, which can be used to calculate the cutoff frequency. The large capacitance means strong electric fields. Thus, the gap between the two ridges serves as the sensing zone. If aqueous sample should be measured, a tube with 100 µm inner diameter goes through it and carries the MUT.

Figure 5.1 (a) Cross view of the ridged section built WR-28 standard waveguide, $W = 0.7$ mm, $G = 0.2$ mm. (b) Equivalent circuit of the ridged waveguide, $L$ is the inductance, $C_d$ the fringe capacitance and $C_s$ the plate capacitance. [5.1] (c) 3D-structure of the ridged waveguide with installing pins and holes and flanges.
The ridge waveguide is fabricated in brass and the 3D illustration is shown in Figure 5.1 (c). The triangle tapering ridge of 25 mm avoids the reflections. A hole of diameter of $D = 250 \, \mu m$ is where the tube goes through. Pins and screws on the body ensure the alignment and connections. Also, in the waveguide machining especially at THz range, the surface roughness should be minimized as much as possible to avoid conduction loss. Huge conduction loss may be caused if the skin depth is at the same order with the surface roughness [5.13]. Moreover, each side of waveguide has standard flanges.

Figure 5.2 (a) Electric field distribution of the ridge waveguide (left) and the standard waveguide (right). (b) Simulation results and the measure results of the ridged waveguide.
Simulations in HFSS [5.14] have been performed to show the electric field distribution and the $S$-parameters. Figure 5.2 (a) is the field distribution of the cross view.

Figure 5.3 (a) Picture of the RF interferometer (b) Broadband measurement of the interferometer.
The electric field of the ridged waveguide is focused between the ridges which significantly increases the detection sensitivity. Due to the tapering, the working band of the can cover 25 GHz to 45 GHz. The simulation and measurement in Figure 5.2 (c) agree well but some ripples are observed in the measurement, which might be caused by the waveguide to coaxial transition adapters.

The RF interferometer is assembled in Figure 5.3 (a). Without a phase shifter, the interferometer can only work at certain frequencies points shown in Figure 5.3 (b). The MUT is 0.1M C₃H₈O (IPA)-water mixture with air as the reference. First, with air $|S_{21}|_{\text{min}}$
is tuned to designated value. Then the IPA solution is injected into the tube. Under both cases, the $|S_{21}|$ is measured. Since the ridged waveguide can work over a wide frequency range, the measurements are also performance at three frequency points.

Measurements results are shown in Figure 5.4. The signal at 25 GHz is smaller than those at higher frequencies, as at higher frequencies, a larger propagation constant $\Delta \beta$ can be induced by the same amount of permittivity change $\Delta \varepsilon$. Also, the response of the IPA solution might be frequency dependent.

TAPERING-WAVEGUIDE SENSOR

As it is shown in our previous work [5.15, 5.16] a filter or a resonator can increase the sensing sensitivity. The permittivity or permeability change of the MUT will affect the passband or the resonant frequency of the sensing devices. The extra induced magnitude change compared with a broadband transmission line can produce higher signal. Waveguide structure is a high pass filter whose passband starting from its cutoff frequency. Therefore, if we work at the cutoff frequencies of the waveguide, the sensitivity improvement principle can be applied, as it is shown in Figure 5.5. Figure 5.6 shows the 3D structure and the cross view of the tapering. The tapering is for impedance matching and avoiding resonance [5.17].
The TE$_{10}$ mode is the dominant mode in such rectangular waveguide [5.2]. As a result, narrowing the sensing zone can increase the cutoff frequency of the waveguide. The
Figure 5.7: Cutoff frequency decreases with the increase of the permittivity of the MUT.

Simulation results in Figure 5.7 agree with the expectation that increased permittivity makes the electrical length longer, therefore, decreasing the cutoff frequency.

**THZ INTERFEROMETER**

In THz range, the size of the waveguides is much smaller than those working at \( K_a \) band. For example, the WR-3 waveguide is in 0.864 mm \( \times \) 0.432 mm. Without using the tapering structures to minimize the sensing zone, the filling factor is still very high. In the THz interferometer, the standard WR-3 waveguide is used as the sensor. Moreover, the power divider is not available in such high frequency range, so instead a modified directional coupler [5.17] is integrated with the waveguide sensor as the power divider. This design also mitigates the connection problems. However, lacking tuning attenuators and phase shifters, the \( f_0 \) and the \( |S_{21}|_{\text{min}} \) is fixed.
One possible solution to decrease $|S_{21}|_{\text{min}}$ is to put a reference material whose permittivity is similar to the MUT in the reference branch. Before the measurement, the same reference material is also put into the measurement branch and then substitute it with the MUT.

Figure 5.8 shows the inside structure of the interferometer and the specific dimensions. In practice, a plate cover forms the top layer and seals the waveguide. There

Figure 5.8 Inner structure and of the THz interferometer. $L_1 = 1.31$ mm and $L_2 = 1$ mm which generates the 180° phase difference. $L_3 = 2.26$ mm, $L_4 = 1.52$ mm, $W_1 = 0.204$ mm, $W_2 = 1.256$ mm.
is a 180° phase difference by the half waveguide wavelength difference between the two branches. The guide wavelength is [5.2],

\[
\lambda_{\text{guide}} = \frac{c}{f} \sqrt{1 - \left(\frac{c}{2a \cdot f}\right)^2}
\]  

(5.1)

The sensing mechanism is associated with the field distribution in a rectangular waveguide. Figure 5.9 shows the electric and magnetic field distribution. Following the field distribution, a piece of MUT in size of 0.1 mm×0.1 mm×0.4 mm is put onto where the electric field or magnetic field is the strongest and a parameter sweep is performed separately to predict the response of the THz interferometer. Similar to the interferometer at lower frequencies, \(\varepsilon'\) and \(\mu'\) causes frequency shift while the \(\varepsilon''\) and \(\mu''\) result in magnitude change, shown in Figure 5.9 (b)-(e).

**REFERENCES**


